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PROCESSING EUTECTICS IN SPACE

(NASA-CR-144196) PROCESSING EUTECTICS IN
SPACE Summary Report (United Technologies
Research Center) 72 p HC \$4.50 CSCL 13H

N76-18183

Unclas

G3/12 18512

by

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SUMMARY REPORT

Contract No. NAS8-29669

June 1973 - November 1975

Prepared for

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 38 35812



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Report R75-911721-10

Processing Eutectics in Space

Summary Report

Contract NAS8-29669

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NO. OF PAGES 69

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Processing Eutectics in Space

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SUMMARY

The investigations of directional solidification made under this contract have indicated the necessity of establishing a secure foundation in earth-based laboratory processing in order to properly assess low-gravity processing.

Work has concentrated on evaluating the regularity of the microstructure of the rod-like eutectic Al-Al₃Ni obtained under different conditions of growth involving the parameters of thermal gradient, solidification rate, and interfacial curvature. In the case of Al-Al₃Ni, where the Al₃Ni phase appears as faceted rods, solidification rate was determined to be a controlling parameter.

It was found very desirable to have a thermal analysis program available in order to design a thermal processing system which would have the desired capabilities to independently vary specific process parameters. During the program herein described, the furnace system was already preexistent. The thermal analysis program, when applied to this furnace, indicated that the interfacial curvature would be constant over a distance of several centimeters within the furnace. This was experimentally verified. It was found that not only was it difficult to change the interfacial curvature between the solid and the liquid during the processing, but it was also difficult to significantly affect the thermal gradient for a fixed ingot diameter. Had the thermal analysis program been fully operative, different configurations of the furnace could have been evaluated to determine how to properly build the furnace system to achieve the desired processability.

Zone melting of thin eutectic films showed that for films of the order of 10 to 20 micrometers thick, the extra surface energy appears to act to stabilize a regular microstructure. In the system examined (the lead-tin eutectic), the lamellae extended from one side of the film to the other in those regions where the film was of the right order of magnitude in thickness. Uniformity of film thickness is required in order to obtain usable results after the zone melt process.

The results of this work suggest that the role of low-gravity as provided in space-laboratory processing of materials is to be sought in the possibility of generating a higher thermal gradient in the solidifying ingot for a given power input-output arrangement than can be obtained under normal one-g processes.

Diffusion controlled processes predominate as solidification rate increases; however, for a eutectic, the upper bound on the rate of solidification is the limiting value of the ratio: (thermal gradient)/(solidification rate). Advantage might be taken of the lack of density-driven convection in a zero-g environment to utilize a low-power furnace system to obtain the same thermal gradient as is provided by a higher powered system in one-g. Alternatively, a higher solidification rate might be achieved.

The data required from zero-g solidification experiments are thus a measure of the thermal gradients achievable under specific furnace operation and solidification conditions, along with an evaluation of the solid-liquid interfacial curvature for comparison with an identical one-g experiment. Conditions should be chosen so that convection would occur in the one-g experiment.

INTRODUCTION

NASA programs have been directed toward exploring the nearly weightless environment provided by orbiting spacecraft to conduct experiments which will lead to manufacturing products in space for use on earth.

It has been recognized that unidirectionally solidified eutectic compositions constitute a class of materials with a high degree of thermal and chemical stability which should be examined in a study of the effects of zero-gravity on material solidification. Only a few of the many eutectic compositions which have been identified and subjected to unidirectional solidification have found potential uses. It is possible that with improved control over the regularity of the phase distribution in a eutectic, additional uses would be found. The role which zero-gravity processing could play in obtaining improved microstructures is presently in question; directional solidification facilities adequate to assess the effect of gravity on eutectic microstructure are not presently available.

In order to define the conditions for a zero-gravity solidification experiment which will provide insight into the role of gravity in affecting the regularity of the eutectic microstructure, an investigation of earth based (one-g) processing of unidirectionally solidified eutectics has been undertaken. The initial study evaluated various aspects of eutectic solidification resulting in a narrowing of the investigation to a study of the processing parameters which control the perfection of the eutectic microstructure. In the conduct of this latter program, the gross curvature of the solid-liquid interface, the rate of solidification, the thermal gradient in the liquid at the solid-liquid interface position, and the specimen shape were examined for their effect on the microstructure.

In addition to the experiments performed to evaluate the effect of these parameters, the NASA-developed SINDA (Systems Improved Numerical Differencing Aalyzer) thermal analysis program was used to calculate the thermal profiles within a solidifying ingot using a model of the laboratory furnace and the thermophysical properties of the eutectic. SINDA is a finite element calculational program with various options, including a corrector subroutine which accounts for phase changes such as melting or solidification. The purpose of this analysis was to aid experimental design of thermal processors to provide the desired degree of experimental control.

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The regularity of the eutectic microstructure was examined using sections transverse to the growth direction which were electropolished and replicated. The replica was photographed at 1200X using an electron microscope, with the resultant plate printed with additional enlargement to obtain a final magnification of approximately 4000X. From the photographs, the (x,y) coordinates of all points were obtained which lay within a 17 x 17 cm square. A computer program was written which operated on all points within a 12 x 12 cm square centered within the larger data field. The distribution of first, second, and third nearest neighbor points was determined; additionally, the angular distribution of the first nearest neighbor points was determined. A high degree of regularity results in these distributions being peaked; the less regular the structure, the more uniform the distribution of neighbor distances and angles.

PROGRAM SEQUENCE

During the first year of the program (June 1973 - June 1974), five areas of investigation were surveyed to assess their appropriateness for extensive development intended to lead to zero-gravity studies. These were:

1. Improvements in structural perfection
2. Study of growth conditions for bending eutectic structures during growth
3. Study of processes for the formation of thin sheets of eutectics
4. Evaluation of floating-zone techniques
5. Processing of off-eutectic compositions.

The results of this study showed that the thermal conditions (temperature distribution and particularly temperature gradient) during the solidification operation are the primary consideration in obtaining planned results and must be subject to a high degree of control. Although the most obvious use of zero-gravity processing of eutectics occurs where convection or other density and/or weight problems such as crucibleless melting led to problems or limitations on earth processed systems, a more important aspect of eutectic usage has to do with the degree of microstructural perfection, or regularity. Many eutectic systems have been studied which might have nonstructural uses if the geometry of the microstructure were more regular.

It was therefore recommended that subsequent work be directed toward determining the effects of thermal conditions during the solidification of model eutectic systems, using the perfection of the microstructure as an evaluative tool for determining the best thermal conditions for specified growth.

Additional recommendations were made to examine the directional solidification of thin eutectic composition films since it had been reported, and verified during the course of the initial investigation, that very uniform structure could be obtained in such a configuration; and that a thermal analysis program should be used to model the experimental thermal conditions in order to suggest modifications which could improve the thermal control.

A six month effort (Modification 2, Contract NAS8-29669, July 1974 - December 1974) was undertaken to set up a furnace-quench thermal processing unit suitable for processing the model eutectics Al-Al₂Cu and Al-Al₃Ni, to examine the directional solidification of thin films of the Pb-Sn eutectic, and to put the SINDA thermal analysis program supplied by NASA-Marshall Space Flight Center into operation at the United Technologies Research Center computing laboratory.

An additional ten month study (Modification 4, Contract NAS8-29669, January 1975 - October 1975) has been conducted during which aligned structure has been obtained in thin films of lead-tin eutectic; microstructural perfection in terms of structural regularity has been assessed as a function of solidification conditions for the model eutectic Al-Al₃Ni; and the SINDA thermal analysis program has been used to compute thermal profiles in the Al-Al₃Ni eutectic during growth by modeling the furnace-quench system.

PROGRAM AREAS OF INVESTIGATION

Bending of Eutectic Structures

The majority of research on directionally solidified eutectics has been carried out by linearly translating a cylindrical ingot through a temperature gradient. Where eutectics are being applied, however, such as for gas turbine components, changing cross sections will force the growth to occur around obstacles in the solidification path. The prototype turbine blade illustrated in Fig. 1 was cast and directionally solidified in one operation. While it is a fairly simple shape, it illustrates the section configurations which are to be expected in practice. In the present investigation, the effect of growth curvature on the microstructure was examined by studying the effects of voids in the ingot on the microstructure of adjacent material, as shown in Fig. 2. It was found that obstacles would disrupt the structure if a shift greater than 45° was required of the structure. Rod-like systems exhibit regions which tend to be devoid of fibers, while plate-like systems are more tolerant.

A NASA-sponsored investigation (Ref. 1) at the United Technologies Research Center (Contract NAS8-27358, August 1972, Final Report) which addressed the use of high-strength, high-temperature eutectic alloys for fastener applications, showed that directionally solidified eutectics could be directly cast into flush-head fastener shapes with adequate shear strengths for actual use. More recently, Garmong (Ref. 2) has examined the structure and crystallography of Al-Al₃Ni and Al-Al₂Cu eutectic alloys solidified in a curved configuration. He finds that the growth direction always lies in the preferred plane forming the interface between the two eutectic phases. The planar atomic-density matching is concluded to be the parameter controlling the interfacial energy in these systems. The fault density appeared to remain constant and similar to that found in linearly solidified specimens.

Off-Eutectic Alloys

Because eutectic compositions are fixed, the structures obtained by directional solidification of these compositions has been considered limited, with only the relative dimensions of the phases adjustable by means of the rate of solidification. However, it has been shown (Ref. 3) that by virtue of solidification in a temperature gradient, composite structure can be obtained even when the composition of the system is significantly off the eutectic composition. This provides an additional degree of freedom in adjusting the composite to obtain specific goals while retaining the desired microstructure.

In Reference 4 a zone-melting technique is described which was used to produce off-eutectic composite structures. This technique was employed in the present study. A high thermal gradient zone furnace was constructed as shown in Fig. 3. The lead-tin system was used to demonstrate the possibility of growing composite structures with controlled microstructure using off-eutectic compositions. The compositions chosen were, by weight, 30% lead - 70% tin (8% deficient in lead), 38.1% lead - 61.9% tin (eutectic), and 45% lead - 55% tin (7% rich in lead). These were passed through the high gradient (330°C/cm) zone furnace at 0.8 cm/hr. The resulting controlled microstructures are illustrated in Figs. 4, 5, and 6. After the controlled solidification experiment, a portion of the specimen with initial composition of 30% lead - 70% tin was examined with the electron microprobe. A quantitative element analysis gave 30.2% lead and 69.8% tin, verifying the identity of initial and final compositions.

The zone-melting technique effectively suppresses convection currents in small diameter samples such as used in this investigation. However, although the compositional constraint is relaxed by employing off-eutectic compositions, additional problems are imposed under conditions where the major part of the liquid above the solidifying ingot is more dense than the boundary layer at the liquid-solid interface. Since the heavier liquid would tend to displace, and therefore disrupt, the boundary layer, off-eutectic solidification studies may be considered candidate experiments to be performed under zero-gravity conditions.

Zone-Melting Techniques

Restricting the size of the molten section of an ingot being directionally solidified provides a means of obtaining high thermal gradients. In addition, it can, if well controlled, be restricted to a sufficiently narrow zone so that the molten material is supported by the surface tension of the material itself, with no requirement for a crucible. It should also be capable of providing a constant rate of solidification, a requirement for obtaining a microstructure with a minimum of faults.

In the present study, the zone melting technique has been experimentally employed in studying the directional solidification of off-eutectic compositions of the lead-tin system, and in the directional solidification of thin films of the lead-tin eutectic alloy. Neither of these studies used a "floating" zone, that is, one with no crucible required. The floating zone, crucibleless processing is used for crystal growing and refining; when other than cylindrical shapes are required, a crucible or shaping container is employed. While it may be possible to preform the material to the desired shape and subsequently zone melt, such an approach has not been studied.

Thin Sheet Form of Eutectics

Many nonstructural materials uses require the material to be in the form of a thin sheet or a surface layer. The direct formation of regular eutectic microstructure in this form has been studied. Guinier, et al (Ref. 5) showed that directional solidification of the lead-tin eutectic could be carried out on sheets of the eutectic with the production of regular, nearly fault-free structure for sheets of thickness 10 micrometers or less. The essentially two-dimensional structure of such a film appears to provide added stability compared to a thick film.

Experiments were performed in which thin films of the lead-tin eutectic formed by rolling cast ingots of the material to thicknesses of the order of 10 to 75 micrometers were zone melted by the heater apparatus illustrated in Fig. 7. The results of these experiments are illustrated in Figs. 8 and 9 which show lamellar structure extending completely through the thickness of the film (Fig. 8) and the tendency of the microstructure to form perpendicular to the surface of the film in thick films (Fig. 9).

Improvements could be made in the regularity of the resulting structure and area of regular structure if uniform thickness films could be formed. A film thickness maximum for the lead-tin eutectic systems appears to be about 20 micrometers to obtain lamellar structure which is continuous from one side to the other.

Microstructural Uniformity - Solidification Conditions

The experiments performed by the astronauts during the manned SKYLAB missions provided a wide variety of preliminary data on materials processing in a low-g environment. Included in the materials processing experiments were solidifications of the aluminum-copper eutectic system. While ingots with regular, controlled microstructure were obtained, the thermal processing conditions were set by the specific combination of materials, and changed with time. Since eutectics have not been developed for purposes where a highly regular microstructure is necessary, proper solidification conditions were not known. Thus, experiments adequately evaluative of the effects of zero-g during solidification could not be well formulated. The studies undertaken in this part of the program were designed to address the relation between the growth conditions and the microstructural uniformity.

Three aspects of the solidification process were chosen for evaluation in terms of their effect on the microstructural uniformity. To help clarify the effects, a furnace was constructed which was intended to provide relatively independent control of the three aspects under study: the thermal gradient, the rate of solidification, and the gross solid-liquid interface curvature.

The furnace used in the present study is illustrated in cross-section in Fig. 10. The main furnace is heated by KanthalTM A-1 wound wire heaters controlled by an on-off control unit. To damp the thermal fluctuations and to provide a uniform thermal environment, a sodium-filled, Inconel 713 heat pipe furnace liner* 25 cm long by 3.5 cm inside diameter was inserted within the cylindrical furnace. The furnace temperature is limited to 1000°C; the maximum operating temperature of the heat-pipe liner is 1100°C. Attached to the bottom of the main furnace is an auxiliary furnace constructed to produce a high power capability source approximately 2.5 cm long by 1.7 cm diameter, sufficient to allow a 1.5 cm diameter aluminum oxide tube to pass through. This auxiliary furnace is located approximately two centimeters below the outlet from the heat pipe. It is constructed from KanthalTM A-1 wire connected to water-cooled copper electrodes. The temperature of this furnace is controlled using an SCR temperature controller with a Pt-Pt/10% Rh thermocouple sensor driving a 16:1 current step-up transformer which can supply up to 200 amperes at 6.3 volts. The nominal power used during solidification experiments is 800 watts for this furnace.

The thermal gradient is established by a quench of flowing water which is sprayed onto the aluminum oxide tube used as a crucible during solidification. The position of the quench relative to the auxiliary furnace can be adjusted. The support rod which is attached to the tube crucible slides through an "O" ring seal in the bottom of the quench apparatus and is attached to a motor driven threaded rod. The drive motor is an adjustable speed DC motor which can span the solidification rate range from 0.2 to 20 cm/hr.

Samples of the aluminum-nickel and aluminum-copper eutectic were directionally solidified using this furnace-quench apparatus at different speeds and with different amounts of insulation below the auxiliary furnace. Changing the quantity of insulation was intended to change the interface position during solidification, and thus the curvature of the solid-liquid interface. The gradient was to be made adjustable by setting the temperature of the auxiliary furnace to different values and adjusting the quench position.

*Dynatherm, Inc., Cockeysville, MD 21030

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A number of studies of the microstructure and crystallography of the aluminum-nickel and aluminum-copper eutectic systems have been published. From Refs. 2,6,7, the aluminum-copper eutectic produces coupled growth with $(1\bar{1}1)_{Al}$ planes parallel to $(21\bar{1})_{Al_2Cu}$ planes. These planes are approximately the Al- Al_2Cu interfacial planes, i.e. the plane described by this condition is uniformly about 6 to 8 degrees from the Al- Al_2Cu interfacial normal. Additionally, the direction normal to the $(011)_{Al}$ planes is parallel to the normal to the $(3\bar{1}3)_{Al_2Cu}$ planes. This direction is uniformly about 9 degrees from the growth direction. The aluminum-nickel system produces faceted rods of Al_3Ni in an aluminum matrix. The crystallographic relations are: the orthorhombic Al_3Ni grows in the $\langle 010 \rangle$ direction in a face-centered cubic aluminum matrix growing in the $\langle 110 \rangle$ direction; the Al_3Ni rods are faceted on the $\{(001)\}$ set of planes, with the facets being parallel to the set of $\{331\}$ planes of the aluminum matrix.

Studies at this laboratory of these two eutectic systems prior to the present work showed that solidification in a temperature gradient with a moving quench and low rate could result in single-grain eutectic rods of the copper-aluminum eutectic (Ref. 8) but produced poor structure in the aluminum-nickel system. These results showed the necessity for improving the control which could be exercised during solidification. Because the aluminum nickel system appeared to be the more difficult system to solidify with good microstructure, more effort was devoted to studying the response of this system than was expended on the aluminum-copper eutectic.

Initial studies of the aluminum-nickel eutectic, determined by zone-melting to have the composition 93.8 w/o Al, 6.2 w/o Ni (Ref. 6), and containing about 11 v/o Al_3Ni rods, were carried out at low rates - of the order of 3 to 5 cm/hr. Typical of the results was an ingot with a high percentage of colony structure, in contrast to the results reported in Ref. 6. Ingot were eventually produced at 5 cm/hr which had no colony structure, but which were characterized by relatively poor regularity. Thermal gradients measured with a thermocouple immersed in an ingot of the aluminum-nickel eutectic could be varied between $50^\circ C/cm$ and $120^\circ C/cm$ at a withdrawal rate of 5 cm/hr. By increasing the withdrawal rate to 12 cm/hr and using the higher gradient, a significant improvement in microstructure was obtained. It would be expected that the thermal gradient would not change significantly due to the change in withdrawal rate, nor should the interface move to any significant extent. Thus, rate of solidification is suggested to be an important parameter controlling the regularity of the microstructure for the Al-Ni eutectic.

An analysis of the rod-structure for regularity is not straightforward, since a "defect" is difficult to define quantitatively. The geometrical regularity of the rod array was chosen as the indicator of quality, with quantitative evaluation specified by determining the nearest neighbor distances and the dispersion of a frequency vs nearest neighbor distance plot. The

frequency vs number of distances for the first twenty nearest distances to a given rod in a transverse section of an ingot was determined. Such a plot includes first, second, and third nearest neighbors, of which there are six each. In a perfect hexagonal array, if the nearest neighbor distance is a , the second nearest neighbors are at a distance of $\sqrt{3} a$, while the third nearest neighbors are at a distance of $2 a$. In addition to this examination, the angular distribution of first nearest neighbors to a given rod in a transverse section was determined. In a perfect hexagonal array, the first nearest neighbors are located at multiples of 60° .

The quantitative examination was carried out by obtaining electron microscope photographs of replicas of electropolished transverse ingot sections. In each case, the largest grain in the section was chosen for examination. The photographs of the replicas were taken at 1200X, and printed with additional magnification to provide a final photograph of the structure at approximately 4000X. The position of the centers of the rods in such a photograph were obtained as (x,y) coordinates on a grid. This information was used as the data file in a computer program (Appendix I) which determined the frequency vs distance and frequency vs angle data from which histograms describing the structure under examination could be constructed.

As indicated in Table I, a number of ingots were directionally solidified to obtain a variety of information, some pertaining to furnace system operation and some specifically for parameter variation. Selected sections of the ingots were examined, and some were chosen for detailed evaluation of the microstructure.

In order to obtain a measure of the parameters being investigated, a solidification experiment was run using a traverse rate of 5 cm/hr with a thermocouple immersed in the melt to measure the thermal gradient, and attempts were made to produce bands (discontinuities in the microstructure at the solid-liquid interface) from which the interface curvature could be determined. Specimen S75-17, an Al-Al₃Ni eutectic, was used for this determination. The crucible was a 13 mm OD aluminum oxide thin-wall (1 mm) Coors Porcelain Co. closed-one-end tube; the length of the ingot was 30 cm. The thermal gradient was measured by a stainless-steel sheathed chromel-alumel thermocouple (.060 in. diameter) protected with an aluminum oxide thermocouple sheathing tube. This was immersed in the melt to the interface position, and then raised in 0.5 cm steps. The thermal gradient thus measured, with the main furnace at 1000°C and the auxiliary furnace at 1000°C maintained by a temperature controller, is 120°C/cm.

A longitudinal section of the ingot showed bands indicating an almost flat solid-liquid interface, as shown in Fig. 11. Normals to the interface bands out to about 70% of the radius make angles with the ingot axis up to about 3.25 degrees. Transverse sections were made at 7.7 cm from the head end, showing

Table I

Ingots Solidified for Structure-Growth Parameter Study

<u>Specimen #</u>	<u>Material</u> Eutectic of	<u>Conditions</u>		<u>Rate</u> cm/hr	<u>Comments</u>
		<u>Main</u> <u>Furnace</u> °C	<u>Auxiliary</u> <u>Furnace</u> °C		
S75-12	Al-Cu	900	600	5.8	50°C/cm
-13	Al-Ni	900	750	4.2	50°C/cm
-14	Al-Ni	1000	975	5.1	Banded nonuniform withdrawal. Stop 10 minute intervals to make bands.
-15	Al-Cu	1000	1000	5	
-17	Al-Ni	1000	1000	5	
-19	Al-Ni	1000	1000	5	Abort
-24	Al-Ni	1000	Ramp*	5	
-26	Al-Ni	1000	Ramp*	5	
-27	Al-Ni	1000	1000	5	G = 120°C/cm
-28	Al-Ni	900	1000	12	Short insulation
-29	Al-Ni	950	1000	12	Longer insulation
-30	Al-Ni	950	1000	12	Ingot remelted
-31	Al-Ni	1000	none	12	Small insulation colony structure

*Ramp down from 1000°C at 10°C/min

colony structure, and at 27.8 cm from the head end, revealing about four grains. Several transverse sections were then made at 1 cm intervals measured from the tail end to examine the microstructure. These sections are shown in Fig. 12. Irregularities in rod size and density indicate that new rods were being produced by branching from existing rods as the growth proceeded. Each branching event causes irregularities in the positioning of the surrounding rods so that the microstructure is irregular in its rod position geometry. Figure 13 shows the best microstructure obtained in ingot S75-17; Figs. 14 and 15 present the structure analysis data.

Two further experiments were then run to determine if varying the input heat from the auxiliary furnace would produce enough solid-liquid interface curvature to cause grains within the ingot to grow rapidly to the surface, thus reducing the total number of grains in the ingot. The first experiment (S75-24) was conducted by reducing the temperature of the auxiliary furnace at a rate of $10^{\circ}\text{C}/\text{min}$ from its initial value of 1000°C when the ingot solidification at 5 cm/hr was started. After an hour, the auxiliary furnace was increased in temperature at the same rate back to 1000°C . The ingot, when sectioned, exhibited colony structure over its complete length. It thus failed to achieve the desired result.

The second experiment (S75-26) paralleled the operation of the first, with the exception that the auxiliary furnace was not turned back on after being lowered in temperature. This ingot exhibited a multiple grain structure, with a slow progression to fewer grains as the tail end is approached. The microstructure is comparable to that obtained in S75-17 discussed above. The progression of grains is shown in Fig. 16. An analysis of the microstructure in several transverse sections is presented in Figs. 17-25.

As noted above, rate of solidification appears to be an important parameter contributing to the production of regular structure in the aluminum-nickel eutectic. Two ingots were solidified at 12 cm/hr with both the main and auxiliary furnaces at stable temperature conditions of approximately 1000°C each. The microstructures of these ingots, S75-28 and S75-29, were analyzed with the results presented in Figs. 26-34. At 12 cm/hr, the microstructures examined are considerably more regular than the microstructures produced by solidification at 5 cm/hr. As the speed of solidification increases, the faceting of the Al_3Ni rods becomes suppressed. A view of the structure of a specimen of $\text{Al}-\text{Al}_3\text{Ni}$ eutectic in transverse section solidified at 100 cm/hr is shown in Fig. 35 where the rods appear circular.

The transition from faceted growth as a controlling mechanism to diffusion controlled growth has not been studied in detail. However, it appears that as the mechanism of diffusion predominates, regularity of the microstructure of a rod-like system should increase.

Thermal Analysis Using the SINDA Program

To provide predictive information relating to the position of the solid-liquid interface in a directionally solidifying ingot, a detailed thermal model of the Bridgman furnace used in this study was constructed and analyzed using the SINDA thermal analysis program obtained from Marshall Space Flight Center. This program will accommodate a three-dimensional model, and provides various subroutines for the analysis, one of which accounts for the change in thermophysical parameters and the latent heat upon phase-change, as occurs with a solidifying system.

Since the program is based on a finite differencing scheme, detailed information is obtained with a large number of node points at the expense of time. After the program was installed on the Research Center's UNIVAC 1110 computer and checked out using problems with known answers, it was determined that the high thermal conductivity of the aluminum-copper and aluminum-nickel eutectics being used would require an inordinate amount of computing time to determine radial temperature profiles with any significant degree of accuracy. Therefore, the number of nodes was decreased in order to determine the position of the solid-liquid interface with reasonable accuracy in a reasonable computing time. It has been determined that using about 300 nodes, distributed along the centerline of the ingot, along the inside boundary of the crucible, and along the outside boundary of the crucible, plus those necessary to specify the input radiating temperatures of various parts of the furnace, resulted in a computer-time of 20 minutes to obtain an accurate location of the solid-liquid interface.

Various values of "adjustable parameters", that is, the heat-transfer coefficients between the aluminum oxide crucible and the furnace (or quench) plus the amount of heat allowed to leak radially through the insulated section, were chosen to attempt to match the calculated thermal profile to the measured thermal profile. The values of the adjustable parameters were constrained to remain within reasonable bounds based on experience. In order to further improve the results of the calculation, a complete thermal profile of the furnace system would have to be undertaken. This effort has been deemed unfeasible under the present study. The results of the thermal calculations are illustrated in Fig. 36; the thermophysical values used for the input to the program are listed in Table II.

For completeness, it is to be noted that thermal transfer by convection is not included in the model used. Although not specifically investigated, it was expected that convection effects would be small because of the high thermal conductivity of the materials used and the lack of inversions in the temperature distribution.

Table II

Thermophysical Properties of System Used for SINDA Calculation

	<u>Thermal Conductivity</u> <u>Kcal/(sec-cm-deg K)</u>		<u>Specific Heat</u> <u>Cp Cal/(gm-deg K)</u>		<u>Density</u> <u>ρ gm/cm³</u>	
	solid	liquid	solid	liquid	solid	liquid
<u>Al-Al₃Ni</u>	0.8	0.2	0.234	0.257	2.81	2.48
6.2 wt/o Ni						
93.8 wt/o Al						
	Heat of fusion 92.6 Cal/gm at the eutectic temp 640°C					
<u>Al-Al₂Cu</u>	0.548	0.2558	0.202	0.243	3.54	3.13
33.3 wt/o Cu						
66.7 wt/o Al						
	Heat of fusion 74.8 Cal/gm at the eutectic temp 548°C					

Al₂O₃ crucible - refer to "Engineering Properties of Selected Ceramic Materials", 1966, Am. Ceramic Society, Columbus, Ohio

SINDA: "Systems Improved Numerical Differencing Analyzer"
Manual 14690-H001-RO-00, Apr. 1971, Ref. 1, 1973, R. L. Dotts
Source - NASA-Marshall Space Flight Center

RESULTS AND DISCUSSION

This investigation has shown that eutectic alloys develop a regular microstructure in response to imposed solidification rate conditions as well as to thermal conditions at the solid-liquid interface. Because of the phase separation upon solidification, the crystallography of the phases requires adjustment of atomic position and size matching in order to minimize the energy of the resulting system. The development of facets on the minor phase is an illustration of the crystallographic requirements. In addition, the rate of solidification constrains the time available for diffusion of the several species in the liquid to the proper position with respect to the growing solid. Hence the finer dispersion of the minor phase at higher solidification rates, and the disappearance of faceting. While it appears that the growth direction is generally parallel to the thermal gradients at the solid-liquid interface, examination of longitudinal sections of directionally solidified eutectics have shown that the expansion of one grain at the expense of its neighbors does not always take place smoothly; rather, jogs in the growth have been seen.

This investigation has shown that for the Al-Al₃Ni eutectic system, growth rate contributes significantly to the development of a geometrically regular microstructure under conditions of constant growth rate, and a curvature of the solid-liquid interface such that the radius of curvature of the interface is greater than 20 times the radius of the ingot. Solidification rates of 12 cm/hr produced significantly better structural results than did solidification rates of 5 cm/hr.

Irregularity of microstructure appears to be associated with the branching of the Al₃Ni rods to generate additional rods as growth proceeds. The introduction of an additional rod requires accommodation of the neighboring rods, and thus a perturbation in geometry. Thermal fluctuations and growth rate fluctuations will therefore perturb the growing microstructure; it is speculated that the perturbation is minimized under conditions of higher solidification rate.

The analysis of the geometry of the rod-like system does not count defects such as nucleation of new rods, or the disappearance of rods; rather, it integrates these effects into the overall geometry by averaging the angular distribution of nearest neighbors of a specified set of rods to determine if, over the areas of examination, a distinct set of symmetry axes can be found which describe the spatial distribution of the rods. An analysis of first, second, and third nearest neighbor distances is also made to determine how distinctly these can be recognized. A more distinct description of a given specimen section could be obtained by initiating the analysis over a relatively small area with a few rods, and compare the results with increasingly larger areas of examination. Small areas could be expected to exhibit greater geometric regularity than large areas in the case of a low degree of overall regularity.

The analysis of the ingot-furnace system using the SINDA thermal analysis program has shown that the analytical scheme can be a powerful tool for aiding in the design of experiments, but that its predictive accuracy is necessarily limited by the detail to which the thermal conditions in the system can be specified. As greater detail is required of the analysis, more accurate information is required for input.

Directional solidification studies on thin eutectic films has been shown to have the potential of producing well-controlled microstructure. The preparation of the initial film has been found to be an extremely important consideration in the production of films with controlled microstructure because, for the thickness levels required (of the order of 10 micrometers), surface tension effects exert forces strong enough to provide mechanical distortions of the films.

CONCLUSIONS AND RECOMMENDATIONS

This study has shown that the degree to which a given furnace and eutectic material system will produce controlled microstructure with a high degree of perfection depends on being able to control the growth rate, the thermal gradient, and the curvature of the solid-liquid interface in the ingot in such a way as to maintain low fluctuations in rate and gradient relative to the growth rate, to maintain an adequate thermal gradient, and to be able to design the furnace to provide the power distribution required to set up the thermal conditions in the ingot which lead to the above described control over the process variables.

It is recommended that further work relating to the improvement of microstructural perfection be transferred from model systems such as the Al-Al₃Ni eutectic to materials of current interest for high-temperature applications, such as the nickel-based eutectic alloy systems. Thermal analysis work should be employed in the design of furnace systems where those are required to develop specific thermal profiles. In any such analysis, the material being processed must be included in computing the resulting thermal profile.

For the planning of space-processing experiments, data from low-gravity experiments should be gathered to determine if steeper thermal gradients can be produced under zero-g conditions than under one-g conditions for the same configuration and material system. Since plane-front eutectic growth is limited by the ratio of (thermal gradient)/(solidification rate), it is desirable to determine if reducing the g-level will automatically result in a steeper gradient. The implications of a positive answer then lie in the area of faster processing and reduction of required power.

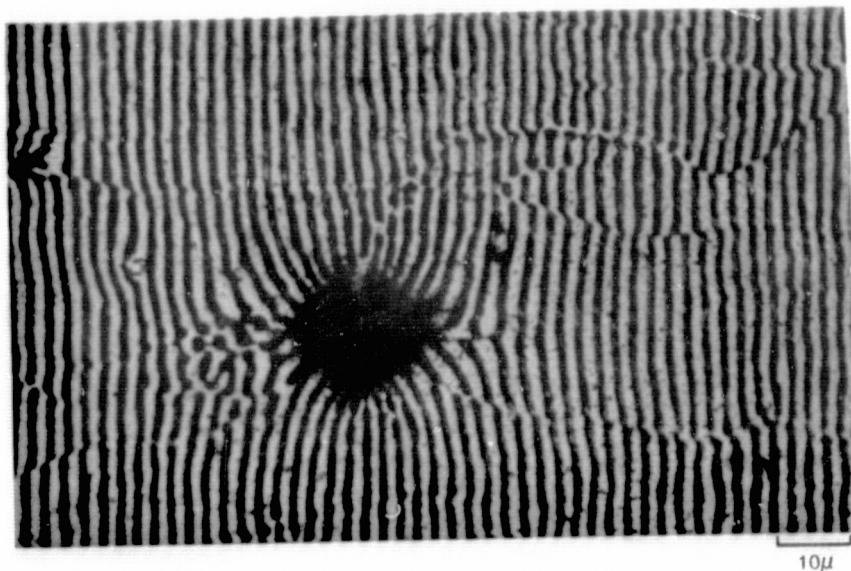
REFERENCES

1. George, F. D. and E. R. Thompson: Final Report, NASA Contract NAS8-27358, August 1972.
2. Garmon, G.: Met. Trans. A, 6A (1975) 1335-1343.
3. Sharp, R. M. and M. C. Flemings: Proceedings of the Conference on In Situ Composites-I, Sept. 1972, NMAB 308-I, Jan. 1973, pp 51 ff.
4. Davis, K. G. and P. Fryzuk: Can. Met. Quarterly 10 (1971) pp 273 ff.
5. Guinier, Pr. A. et al: Proceedings of the Conference on In Situ Composites-I, Sept. 1972, NMAB 308-III, Jan. 1973, pp 85 ff.
6. Lemkey, F. D., R. W. Hertzberg and J. A. Ford: Trans. Met. Society of AIME, 233 (1965) 334-341.
7. Kraft, R. W. and D. L. Albright: Trans. Met. Society of AIME, 221 (1961) 95-102.
8. George, F. D. and F. Galasso: Preparation of Single Grain Eutectics for the M 566 Experiment, Modification 2, Contract NAS8-28724, Dec. 15, 1972.

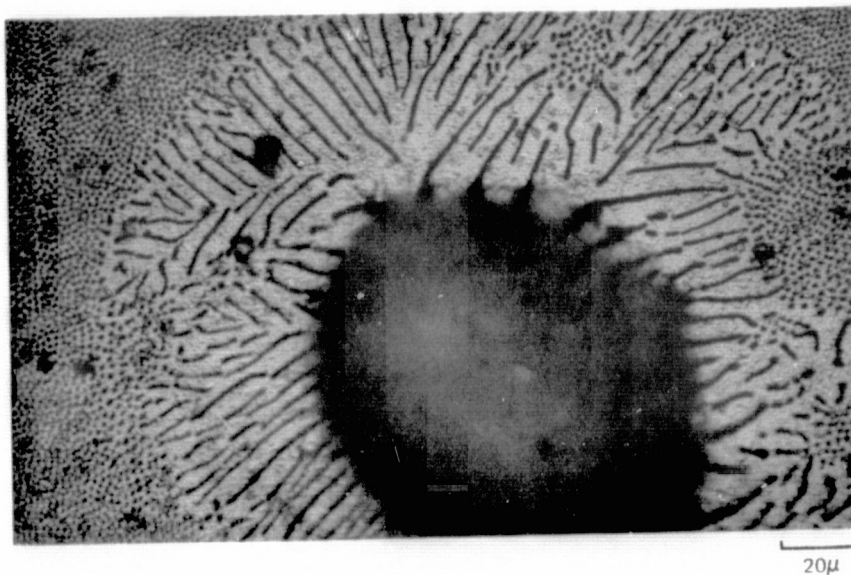


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FIGURE I. DIRECTIONALLY SOLIDIFIED Ni-Cb-Al HIGH TEMPERATURE EUTECTIC ALLOY
LONGITUDINAL SECTION



LAMELLAR Al-Al₂Cu TRANSVERSE SECTION THROUGH INTERNAL VOID



RODLIKE Al-Al₃Ni TRANSVERSE SECTION THROUGH INTERNAL VOID

FIGURE 2. ACCOMMODATION OF EUTECTIC MICROSTRUCTURE
TO REGIONS REQUIRING CHANGES IN GROWTH DIRECTION

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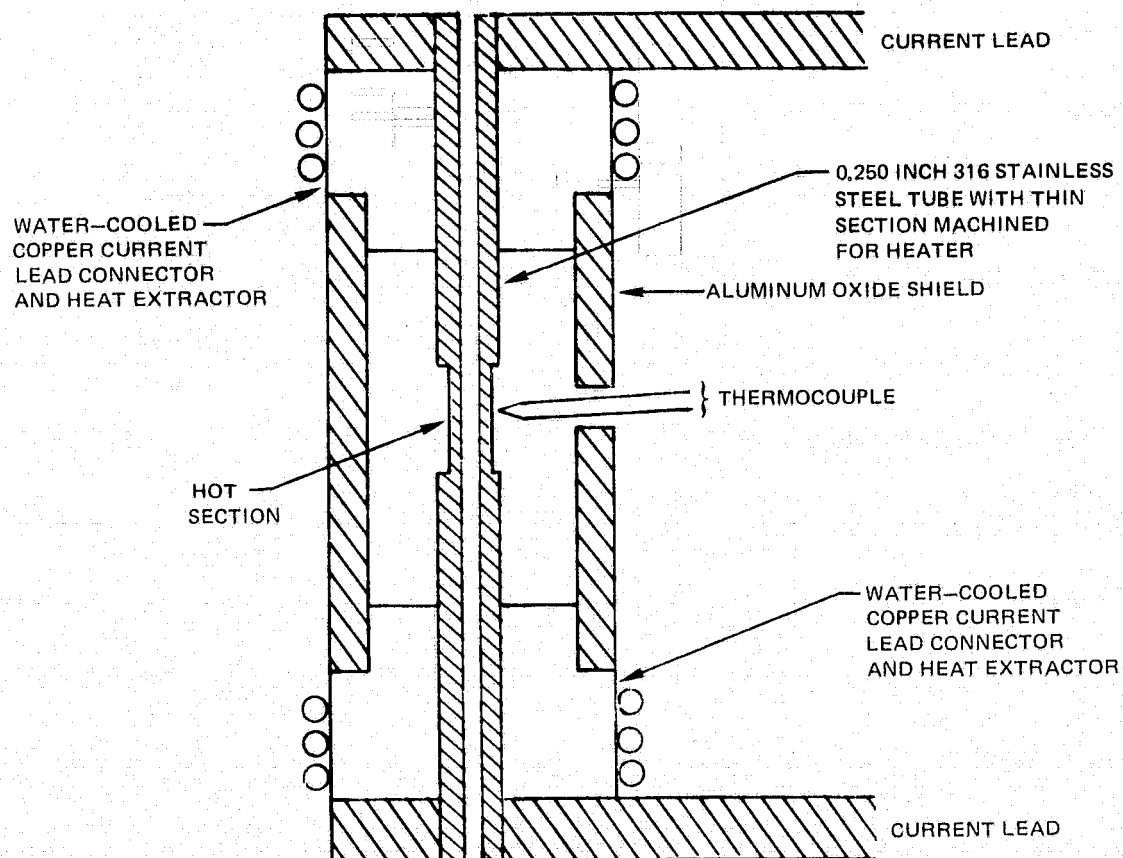
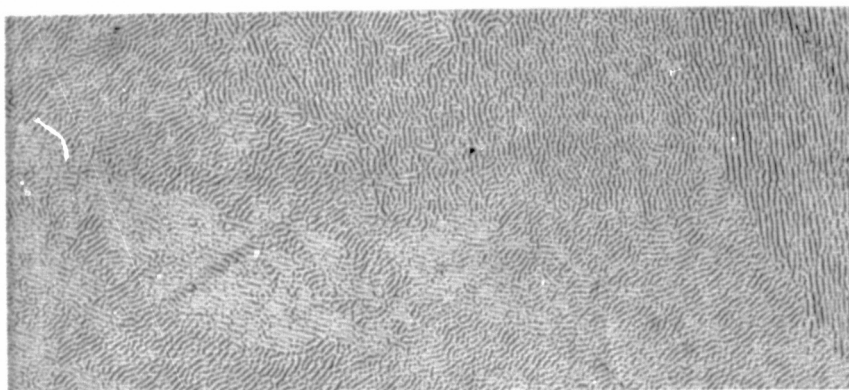


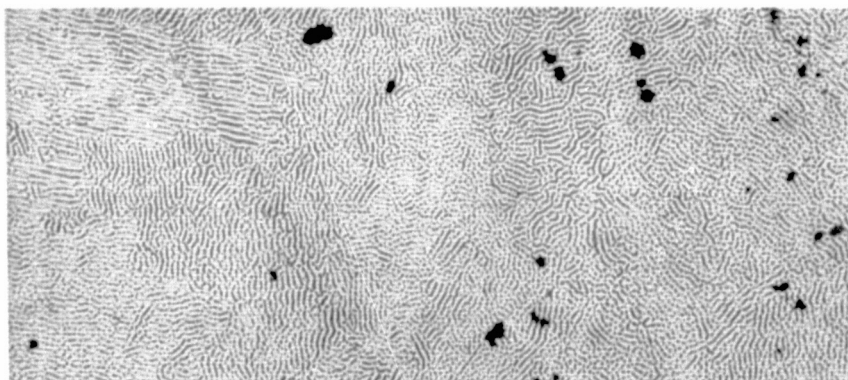
FIGURE 3. HIGH THERMAL GRADIENT ZONE FURNACE

UDS 721-41 330°C/cm @ 0.8 cm/hr
(EUTECTIC: 38.1% LEAD)



INITIAL CONTROLLED REGION

100μ



2 cm INTO CONTROLLED REGION

100μ

FIGURE 4. MICROSTRUCTURE IN 30% LEAD-70% TIN
ZONE DIRECTIONALLY SOLIDIFIED OFF-EUTECTIC ALLOY

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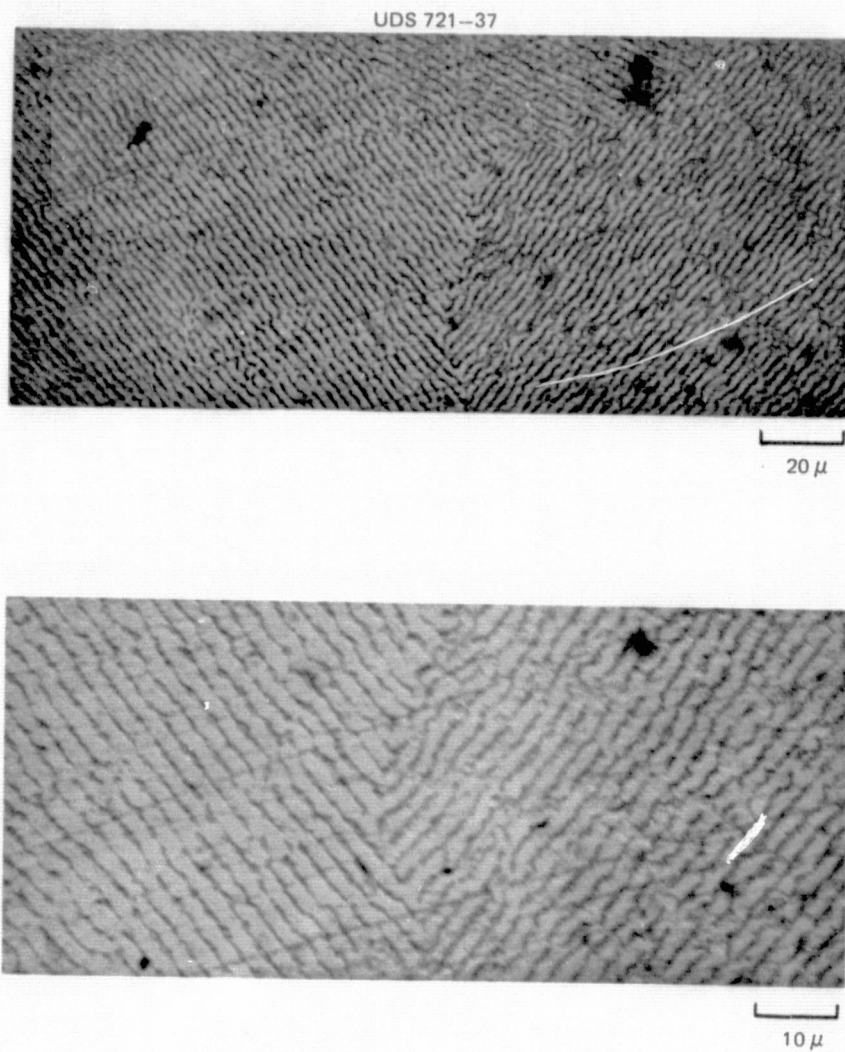
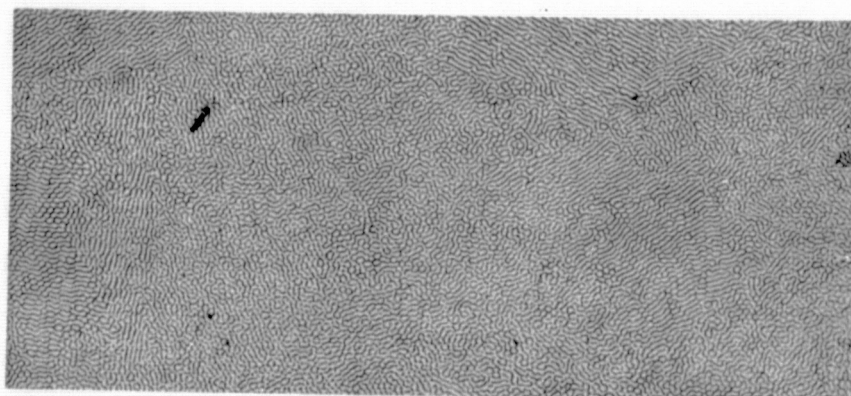


FIGURE 5. MICROSTRUCTURE OF LEAD-TIN EUTECTIC ZONE MELTED
AT 3.4 cm/hr WITH A THERMAL GRADIENT OF 165°C/cm

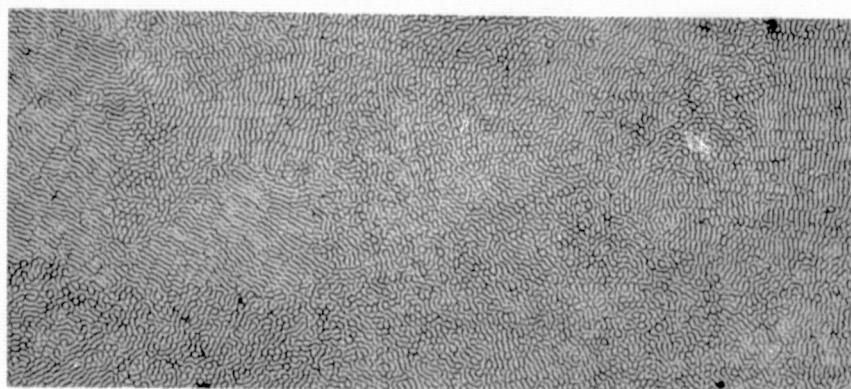
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UDS 721-40 330°C/ cm @ 0.8 cm/hr
(EUTECTIC: 38.1% LEAD)



HEAD END

100μ



6 cm PAST HEAD END

100μ

FIGURE 6. MICROSTRUCTURE IN 45% LEAD-55% TIN
ZONE DIRECTIONALLY SOLIDIFIED OFF-EUTECTIC ALLOY

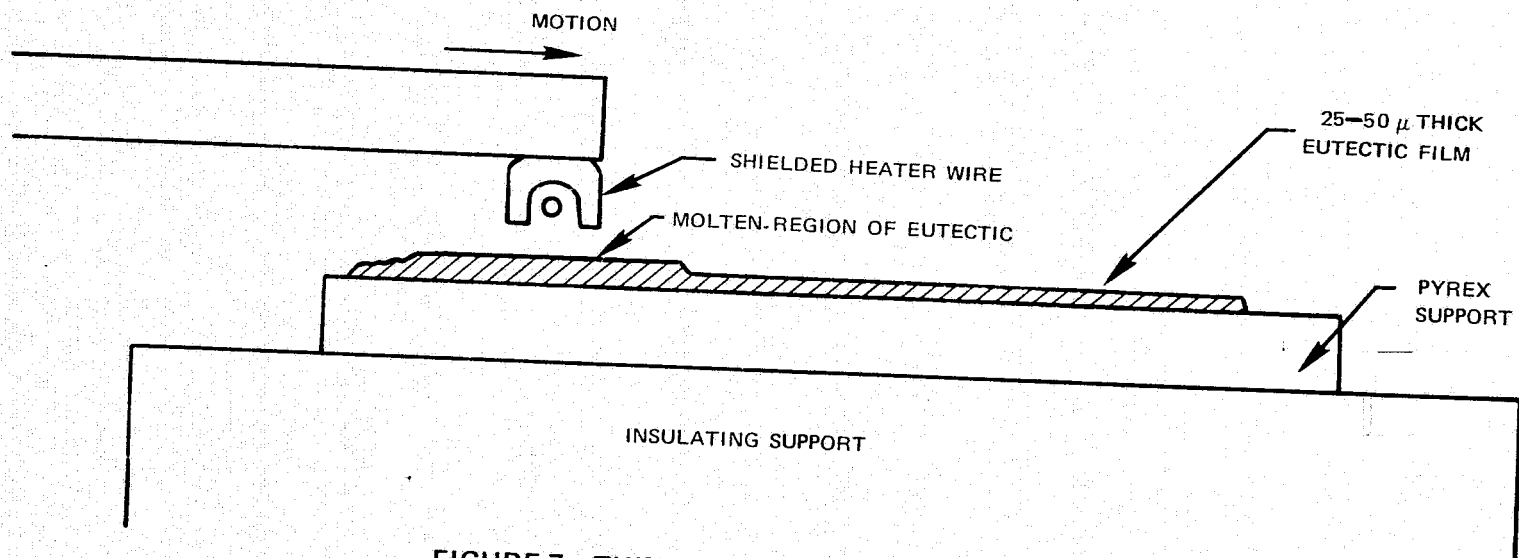


FIGURE 7. THIN SHEET ZONE MELTING APPARATUS

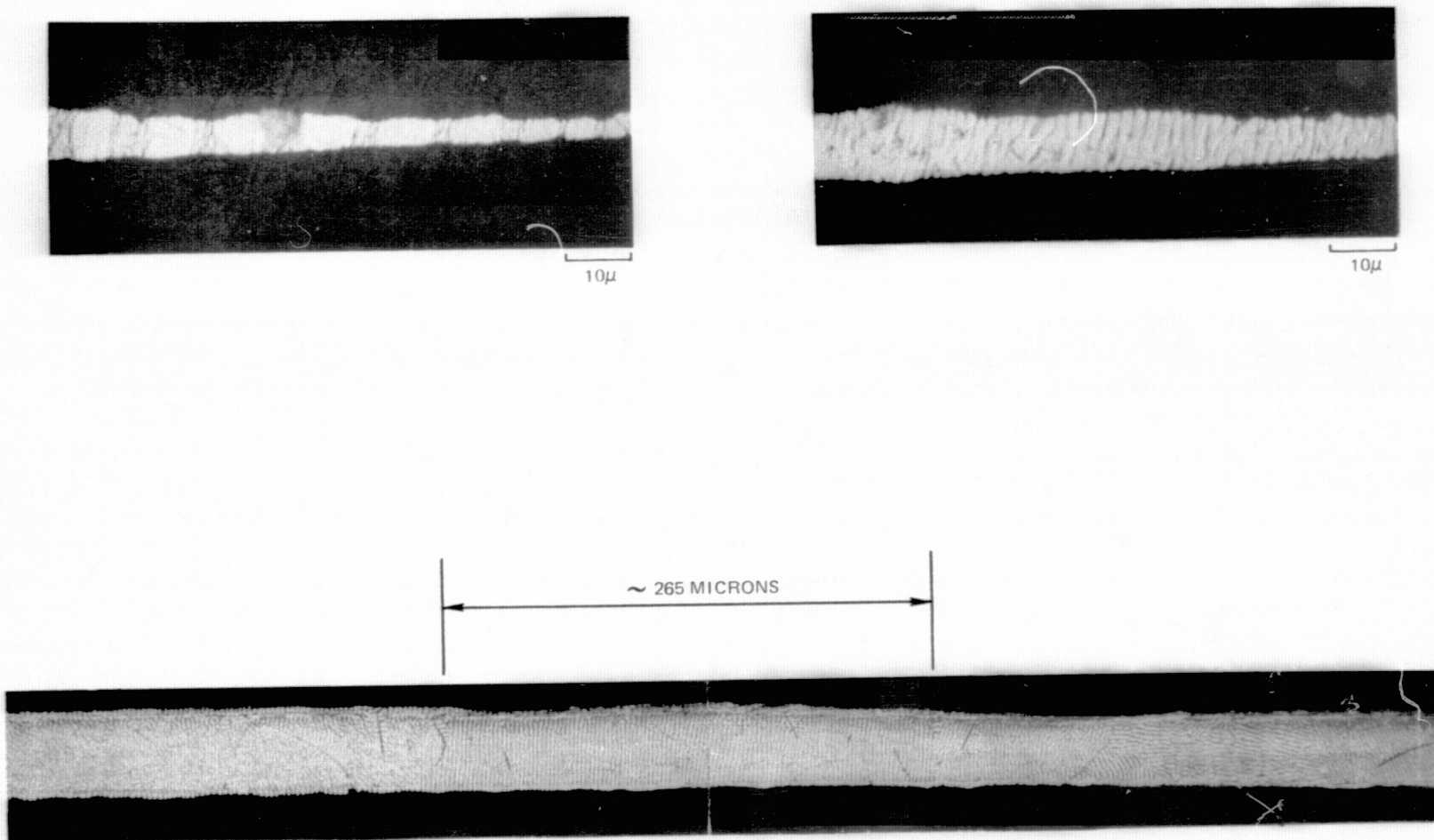


FIGURE 8. TRAVELING-ZONE-PROCESSED LEAD-TIN EUTECTIC FILM 45 MICRONS THICK
SHOWING ALIGNED LAMELLAR MICROSTRUCTURE THROUGH COMPLETE FILM
(SECTION TRANSVERSE TO PROCESS DIRECTION)

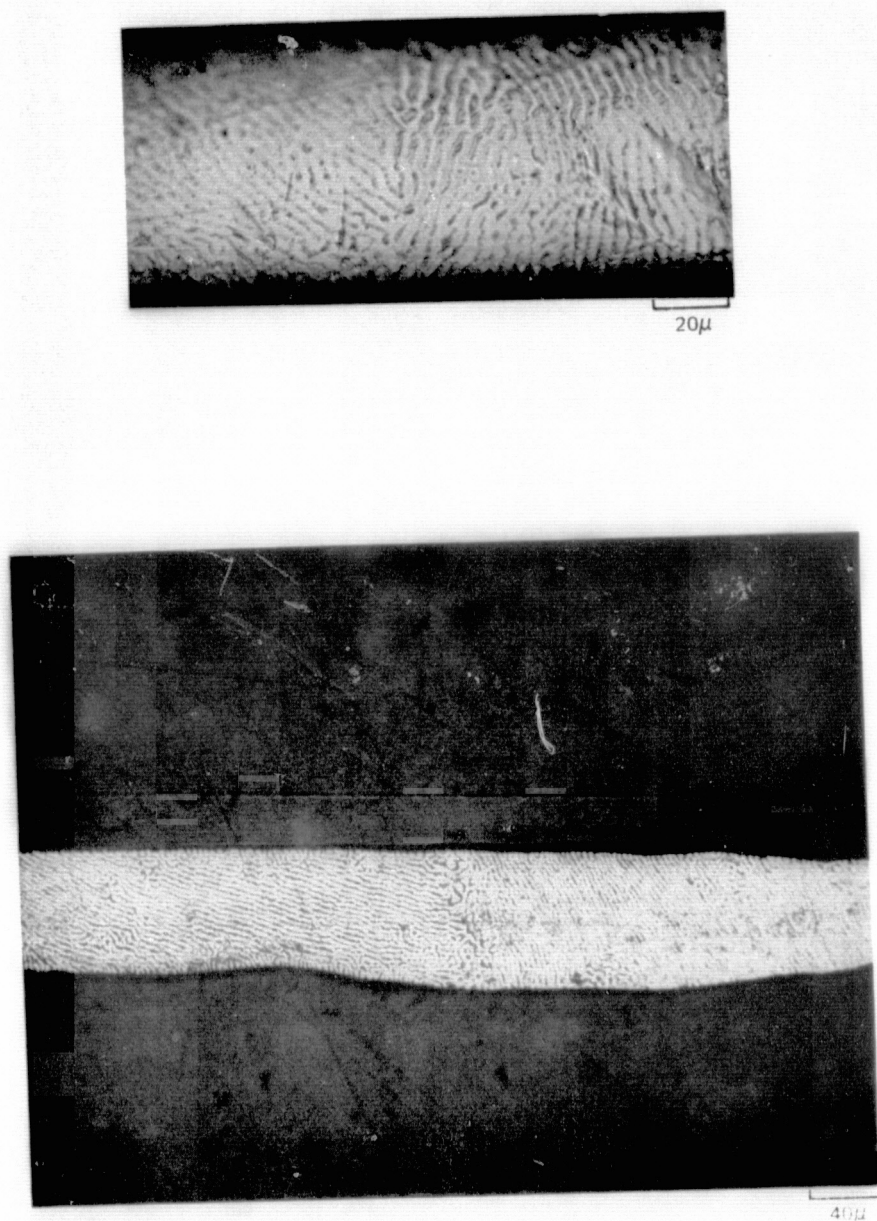


FIGURE 9. TRANSVERSE SECTIONS OF THE THICK PORTIONS OF LEAD-TIN EUTECTIC FOILS
SHOWING LAMELLAR ORIENTATION AT THE FOIL SURFACES

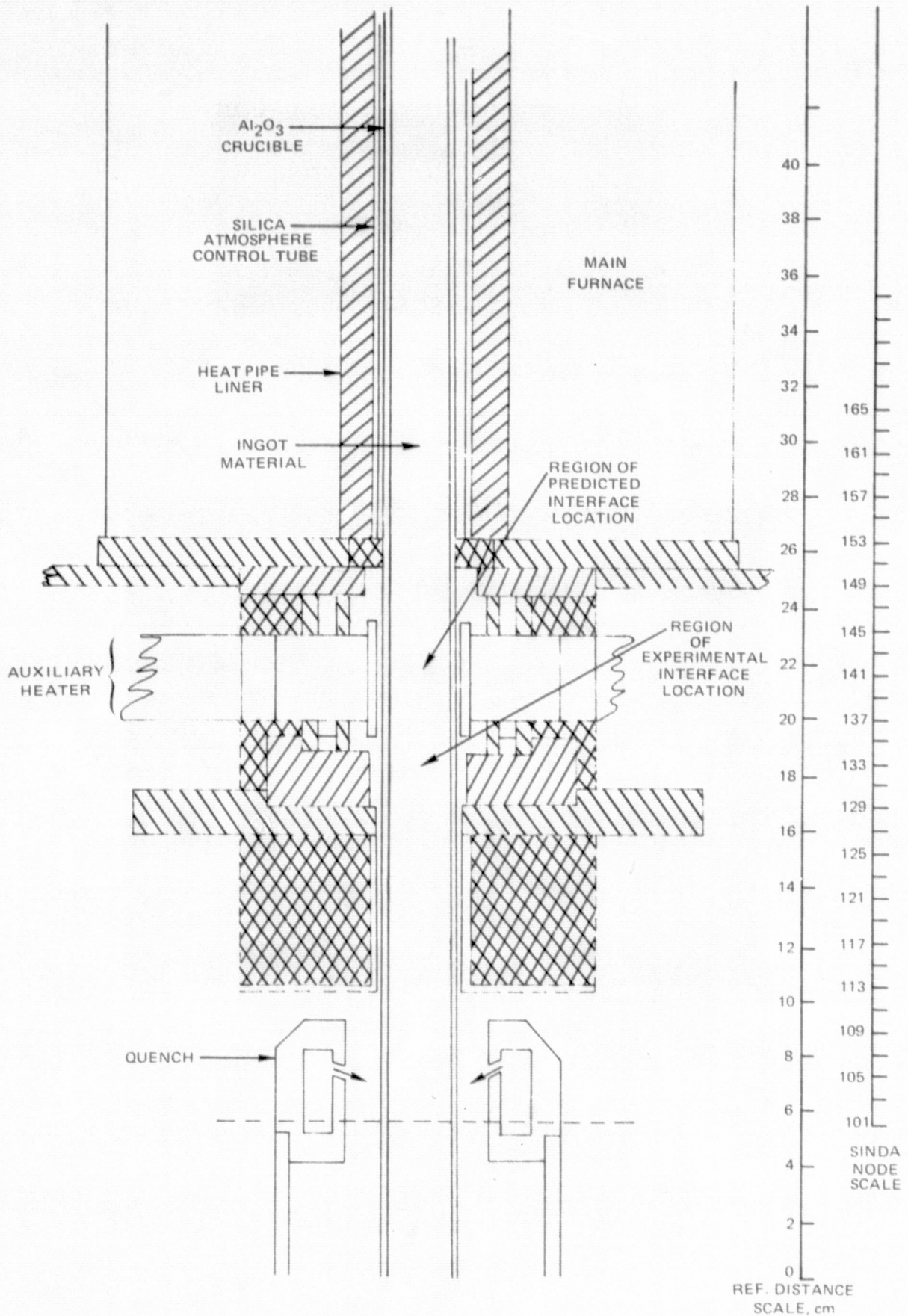


FIGURE 10. SECTION THROUGH BRIDGEMAN FURNACE CONFIGURATION

FURNACE TEMP: 1000°C RATE: 5 cm/hr

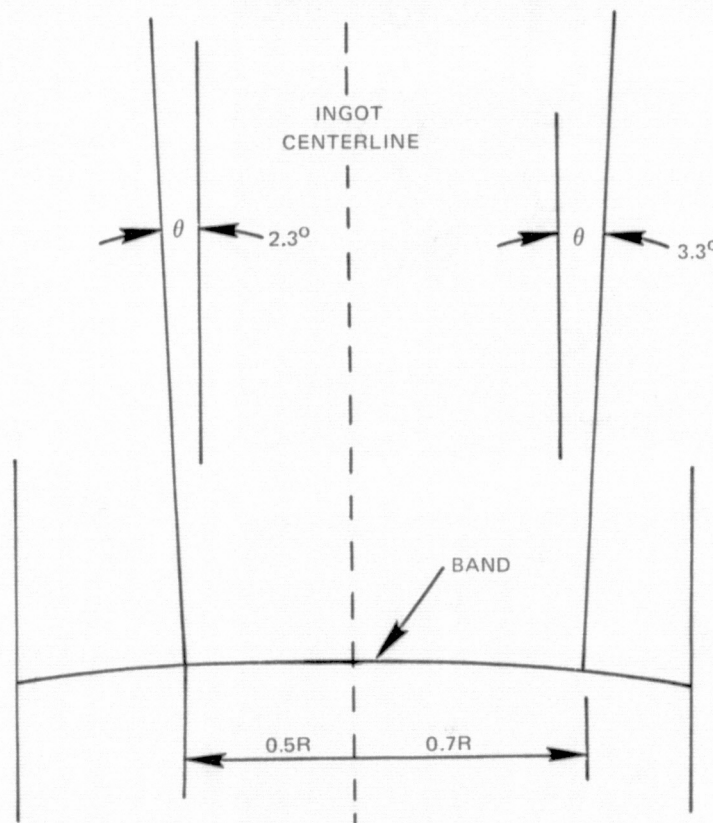
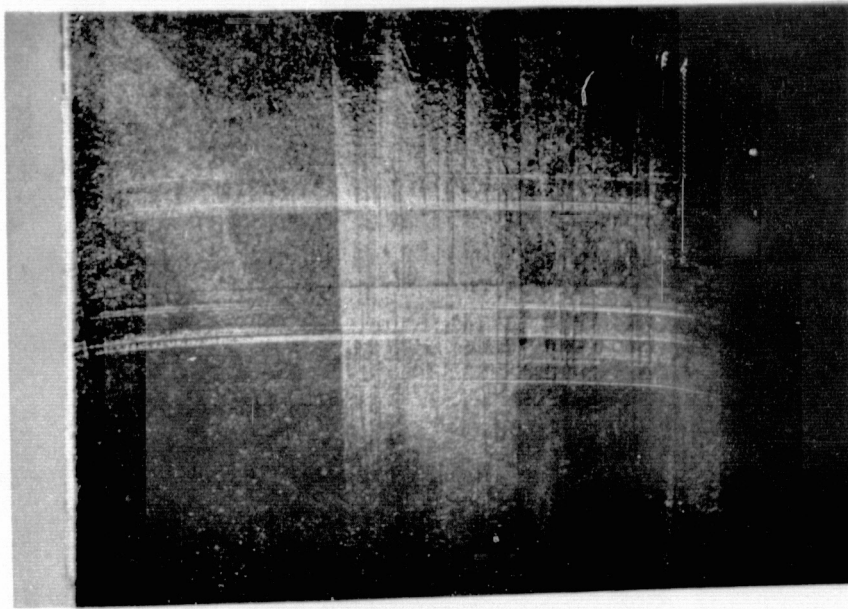
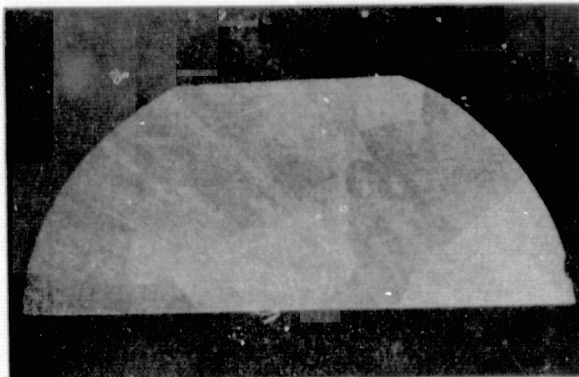
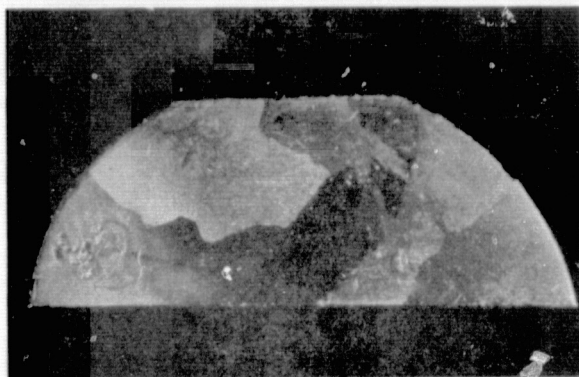


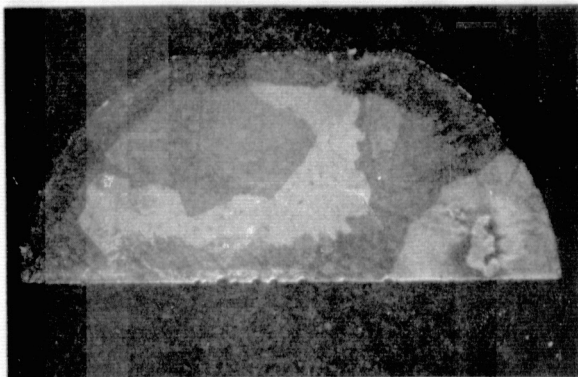
FIGURE 11. LONGITUDINAL SECTION OF INGOT S75-17 SHOWING BANDS GENERATED TO REVEAL SOLID-LIQUID INTERFACE CURVATURE



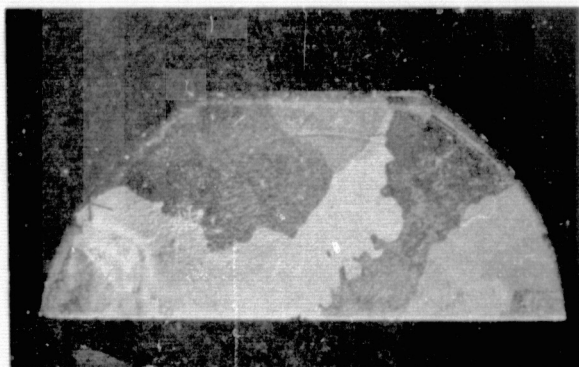
110mm FROM HEAD



100mm FROM HEAD



90mm FROM HEAD



80mm FROM HEAD

FIGURE 12. TRANSVERSE SECTIONS OF INGOT S75-17 SHOWING THE CHANGES IN GRAIN SIZE; RATE 5 CM/HR.

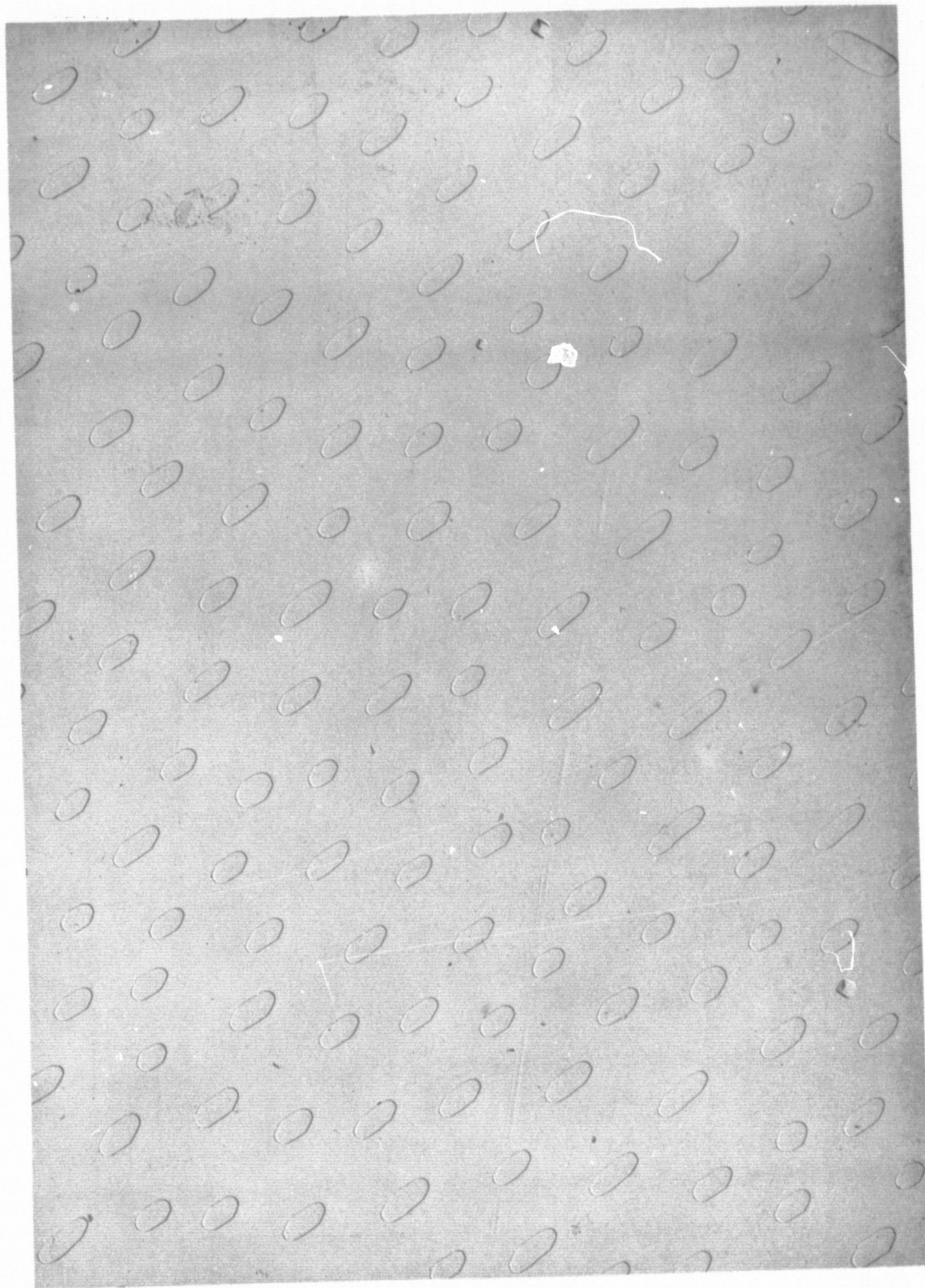


FIGURE 13. Al-Al₃Ni EUTECTIC SPECIMEN S75-17; REPLICA OF TRANSVERSE SECTION;
MAGNIFICATION 8400X; RATE 5cm/hr

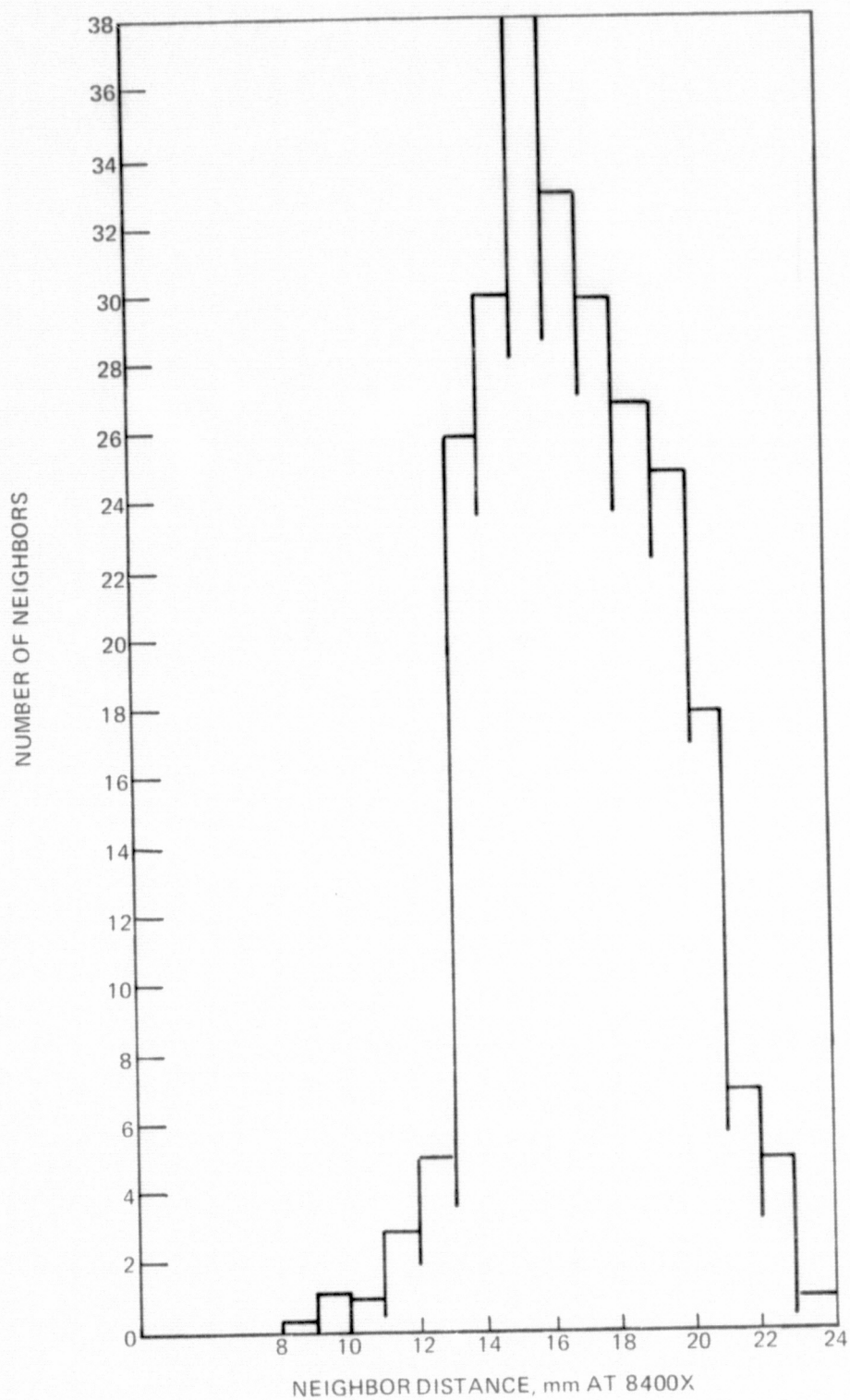


FIGURE 14. DISTRIBUTION OF NEAREST NEIGHBOR DISTANCES FOR SPECIMEN S75-17 Al-Al₃ Ni EUTECTIC FROM TRANSVERSE SURFACE REPLICA

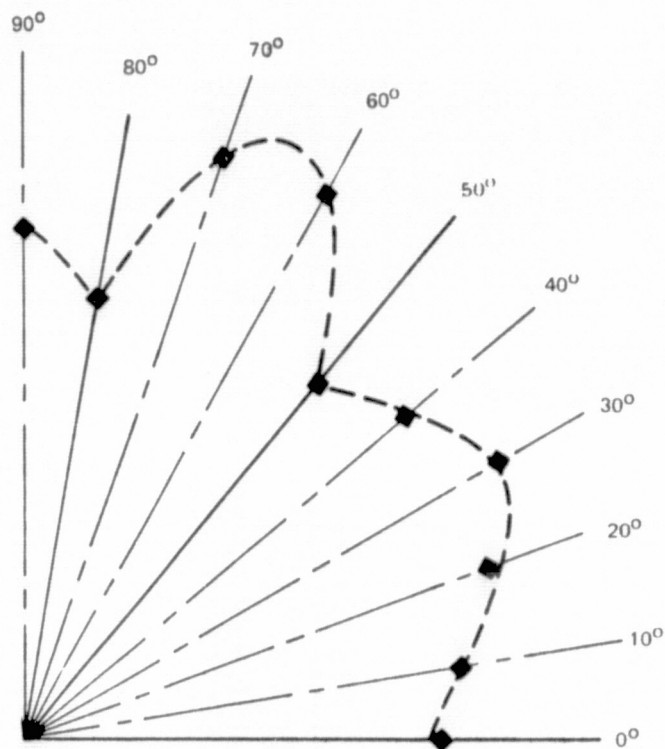
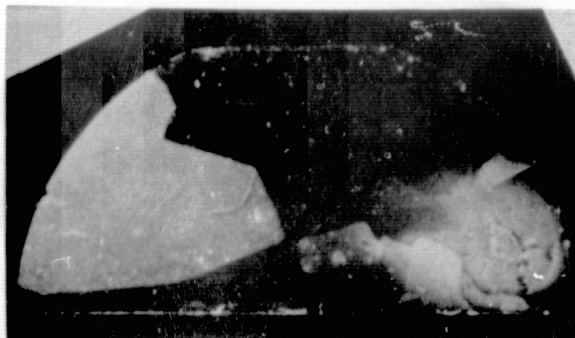
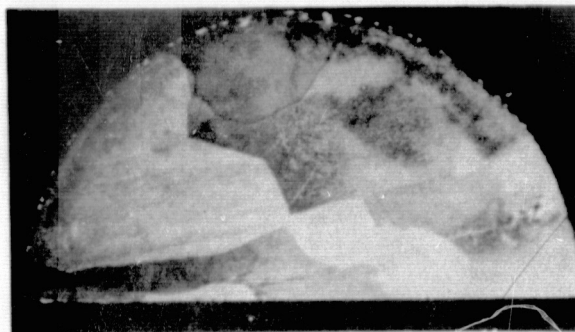


FIGURE 15. ROSE-OF-THE-NUMBER-OF-INTERSECTION PLOT FOR Al-Al₃Ni EUTECTIC SPECIMEN S75-17 FROM INTERSECTION COUNTS AT 10° INTERVALS SHOWING APPARENT ORIENTATION AXES FOR THE FIRST 90°



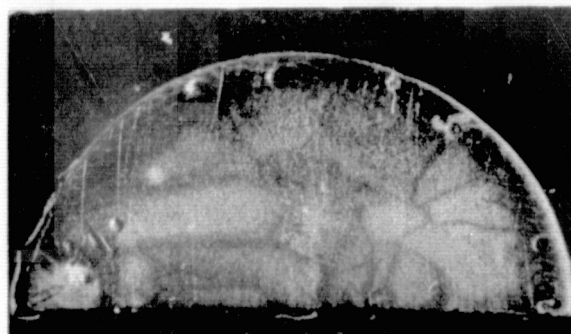
160 mm FROM HEAD



150mm FROM HEAD



140mm FROM HEAD



130mm FROM HEAD

FIGURE 16. TRANSVERSE SECTIONS OF INGOT S75-26; SHOWING THE CHANGES IN GRAIN SIZE; RATE 5cm/hr

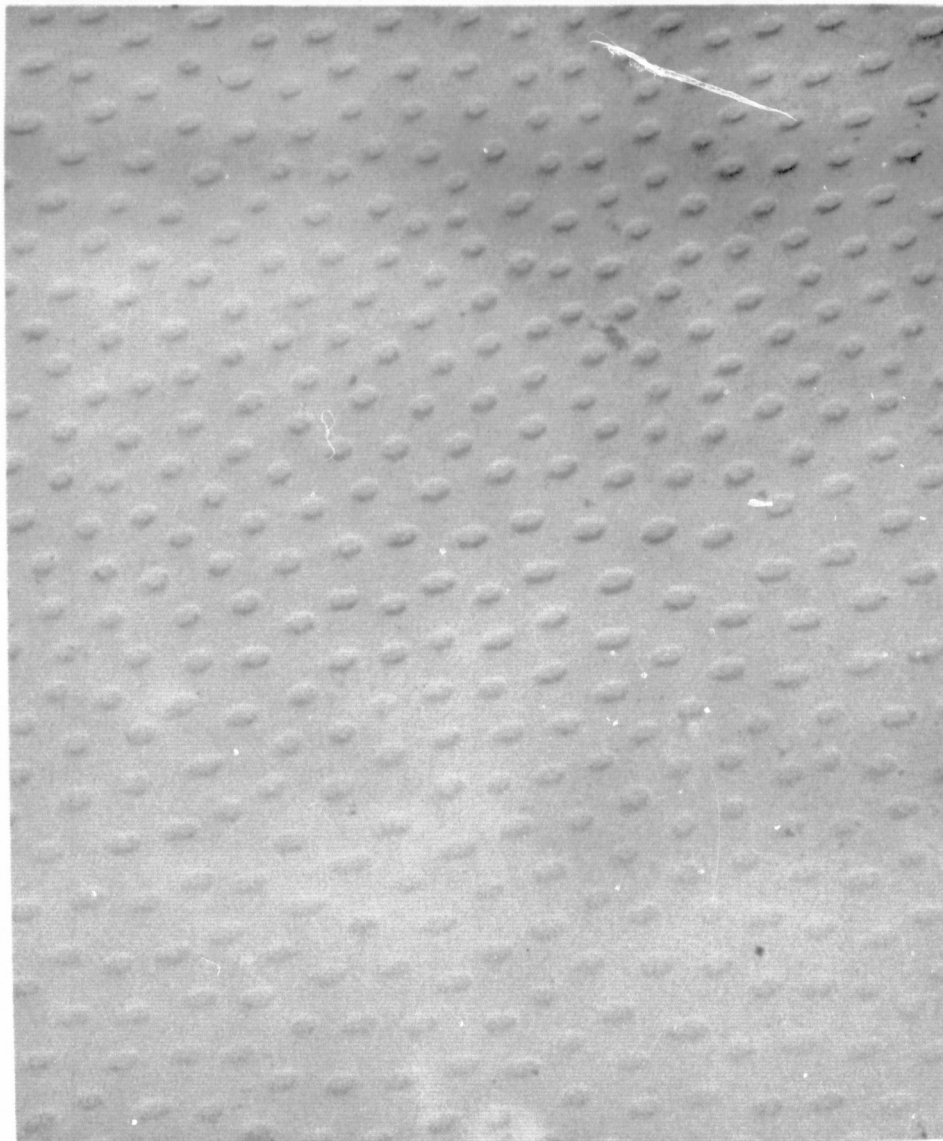


FIGURE 17. Al-Al₃Ni EUTECTIC SPECIMEN S75-26; REPLICA OF TRANSVERSE SECTION 6 ; MAGNIFICATION 3800X ; RATE 5 cm/hr

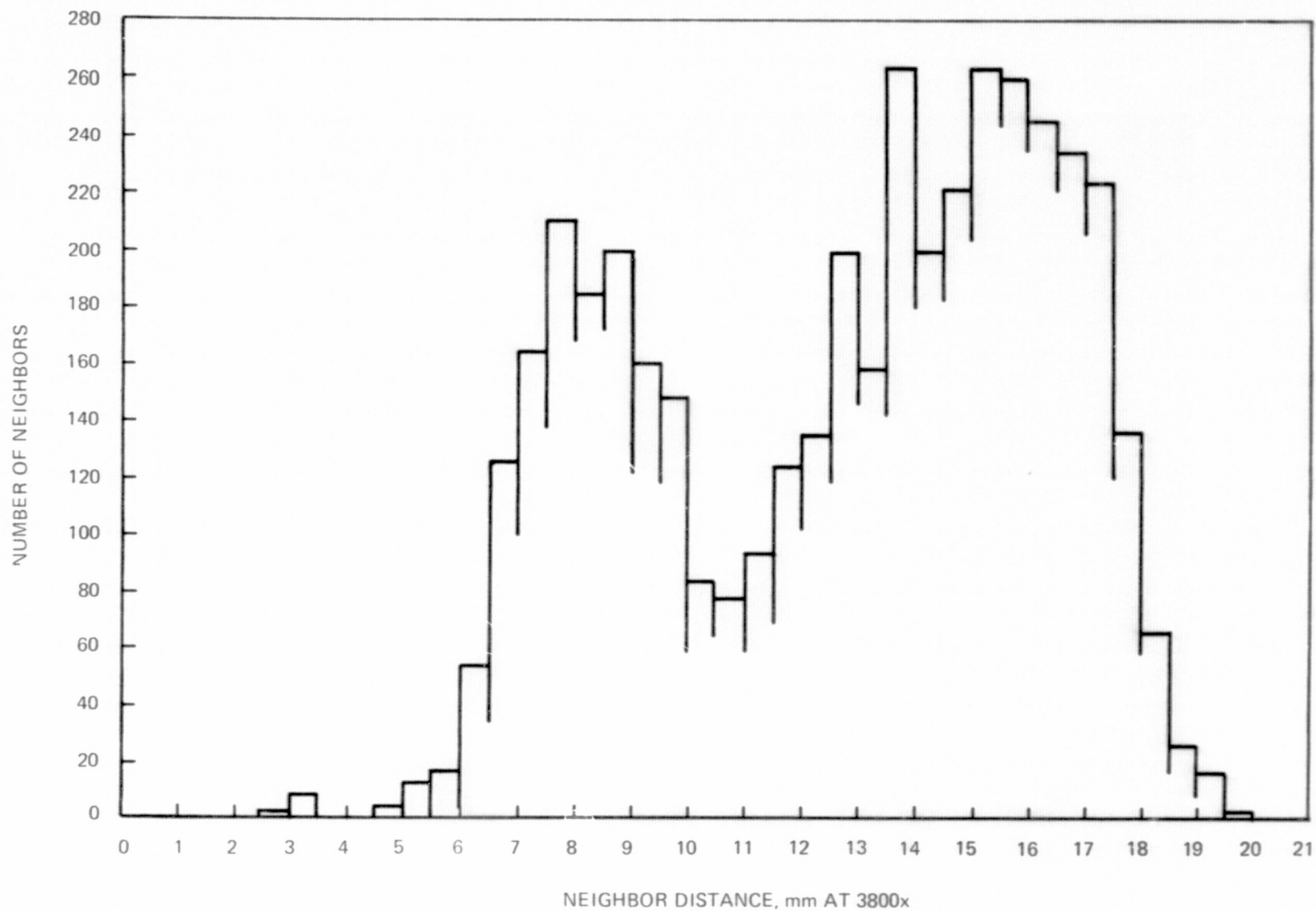


FIGURE 18. FIRST, SECOND, AND THIRD NEAREST NEIGHBOR DISTRIBUTION FOR Al-Al₃ Ni EUTECTIC
SPECIMEN S75-26 #196; 5 cm/hr; DATA BASE 240 POINTS

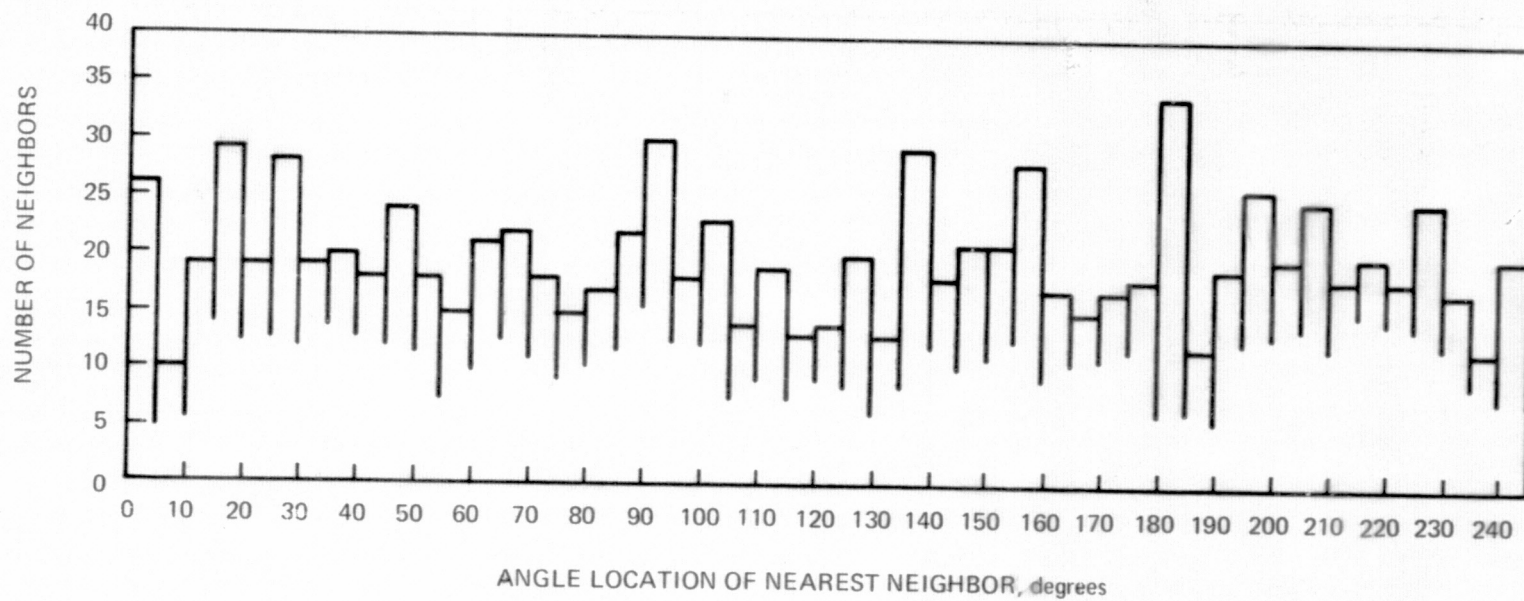
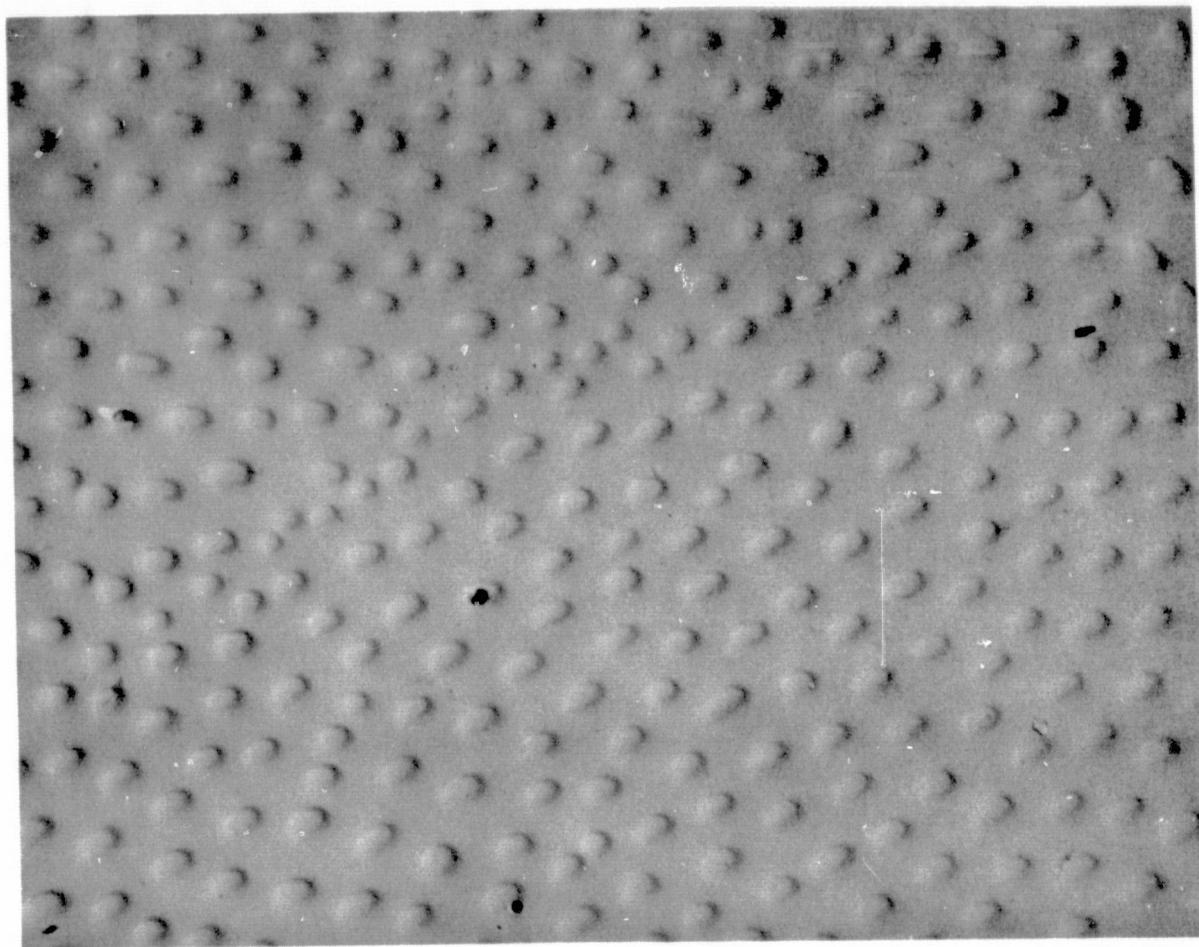


FIGURE 19. NEAREST NEIGHBOR ANGULAR DISTRIBUTION FOR Al-Al₃Ni EUTECTIC SPECIMEN S75-26 #196;
5 cm/hr; DATA BASE 240 POINTS



3800X

FIGURE 20. Al-Al₃Ni EUTECTIC SPECIMEN S75-26; REPLICA OF SECTION 7;
MAGNIFICATION 3800X; RATE 5cm/hr

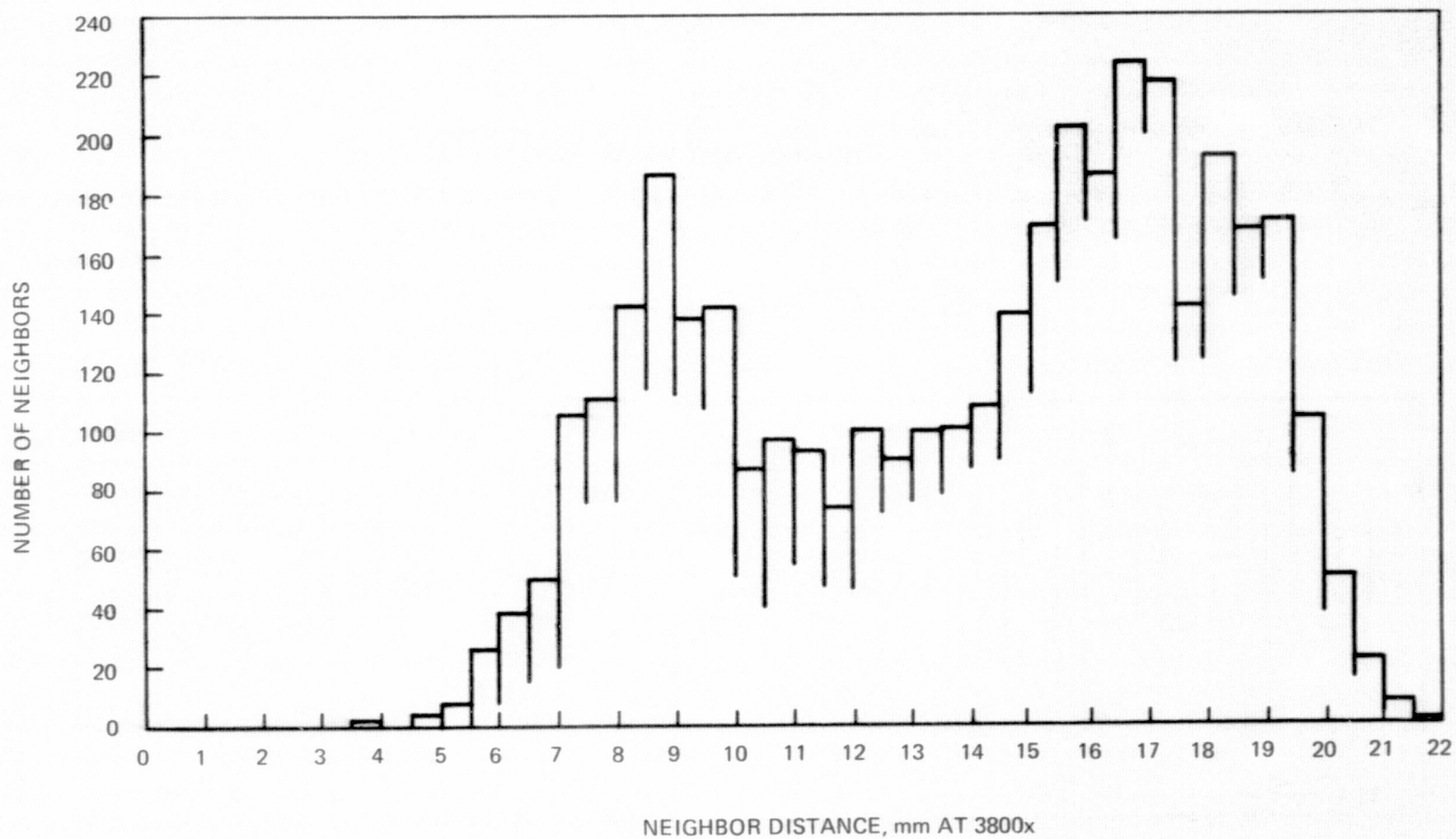


FIGURE 21. FIRST, SECOND, AND THIRD NEAREST NEIGHBOR DISTRIBUTION FOR $\text{Al}-\text{Al}_3\text{Ni}$ EUTECTIC
SPECIMEN S75-26 #197; 5 cm/hr; DATA BASE 211 POINTS

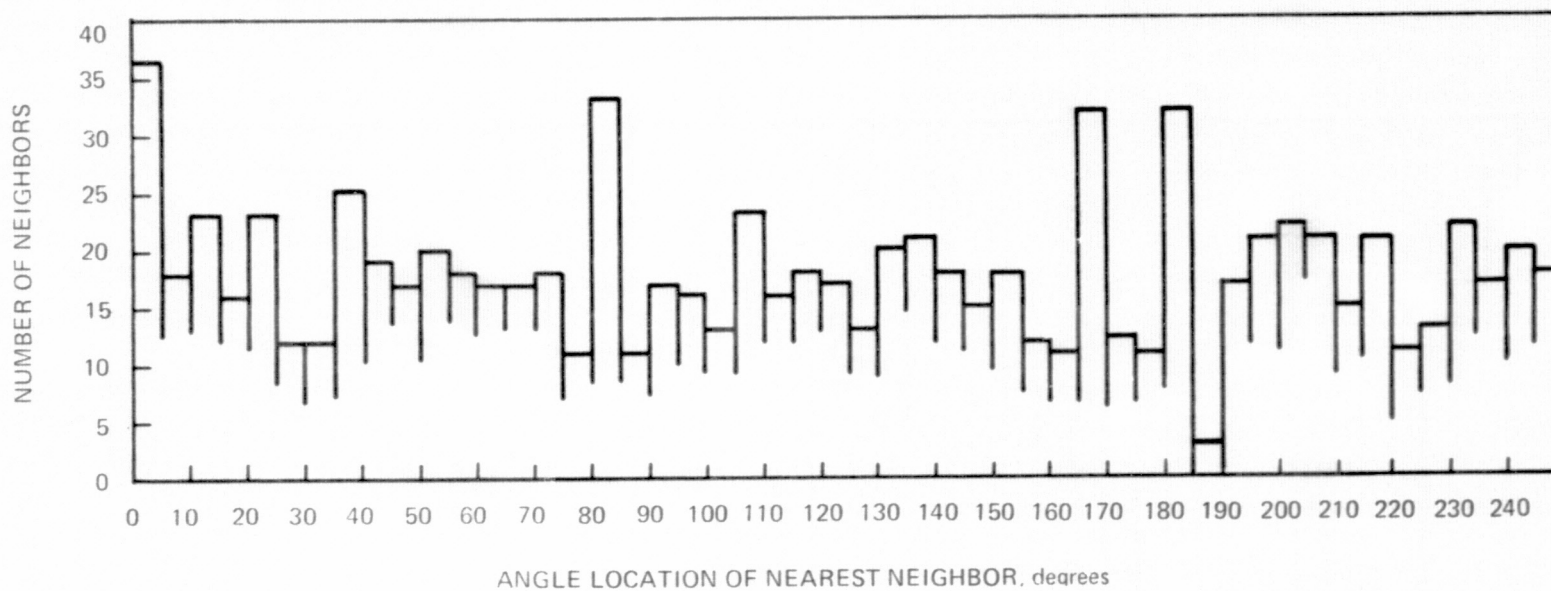
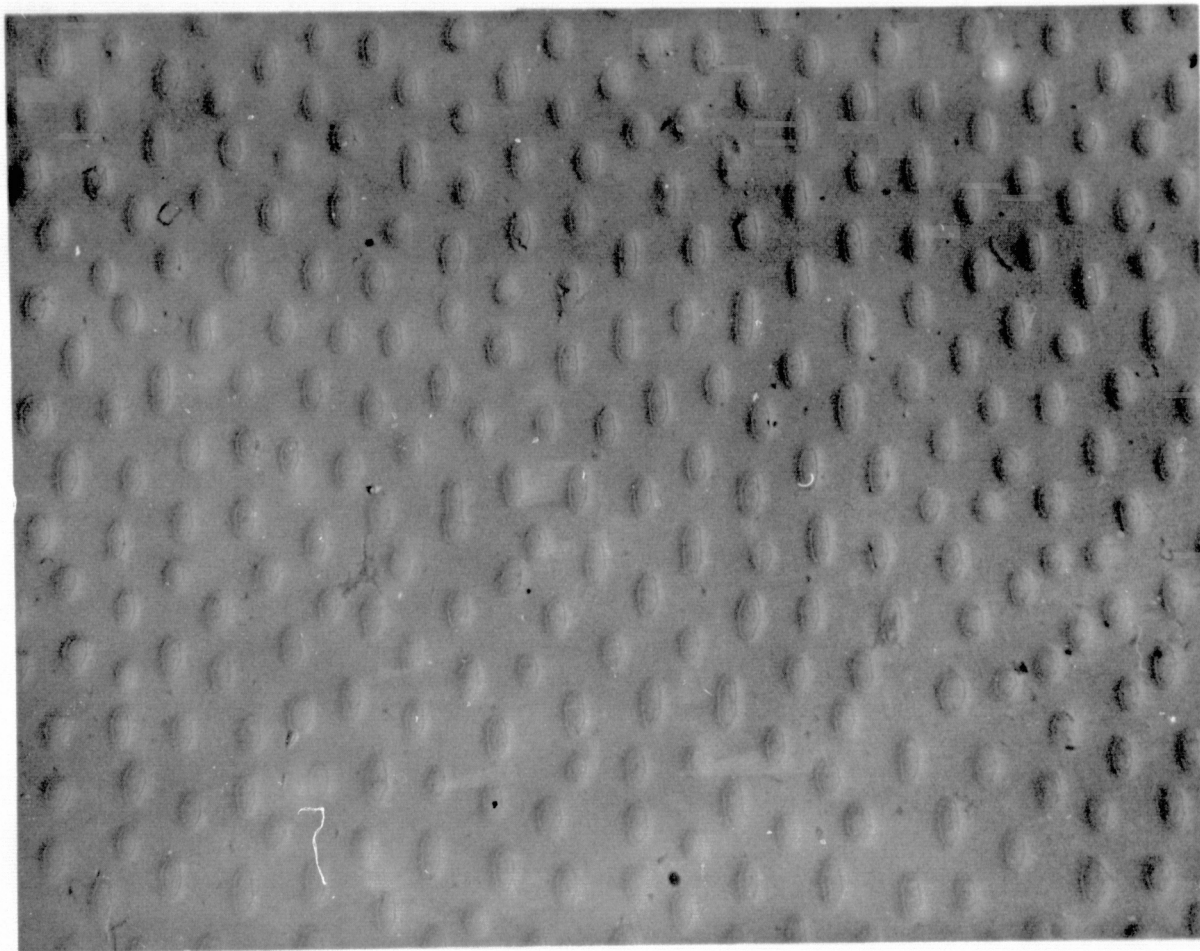


FIGURE 22. NEAREST NEIGHBOR ANGULAR DISTRIBUTION FOR Al-Al₃Ni EUTECTIC SPECIMEN S75-26 #197;
5 cm/hr; DATA BASE 211 POINTS



3800X

FIGURE 23. Al-Al₃Ni EUTECTIC SPECIMEN S75-26; REPLICA OF SECTION 8;
MAGNIFICATION 3800X; RATE 5 cm/hr

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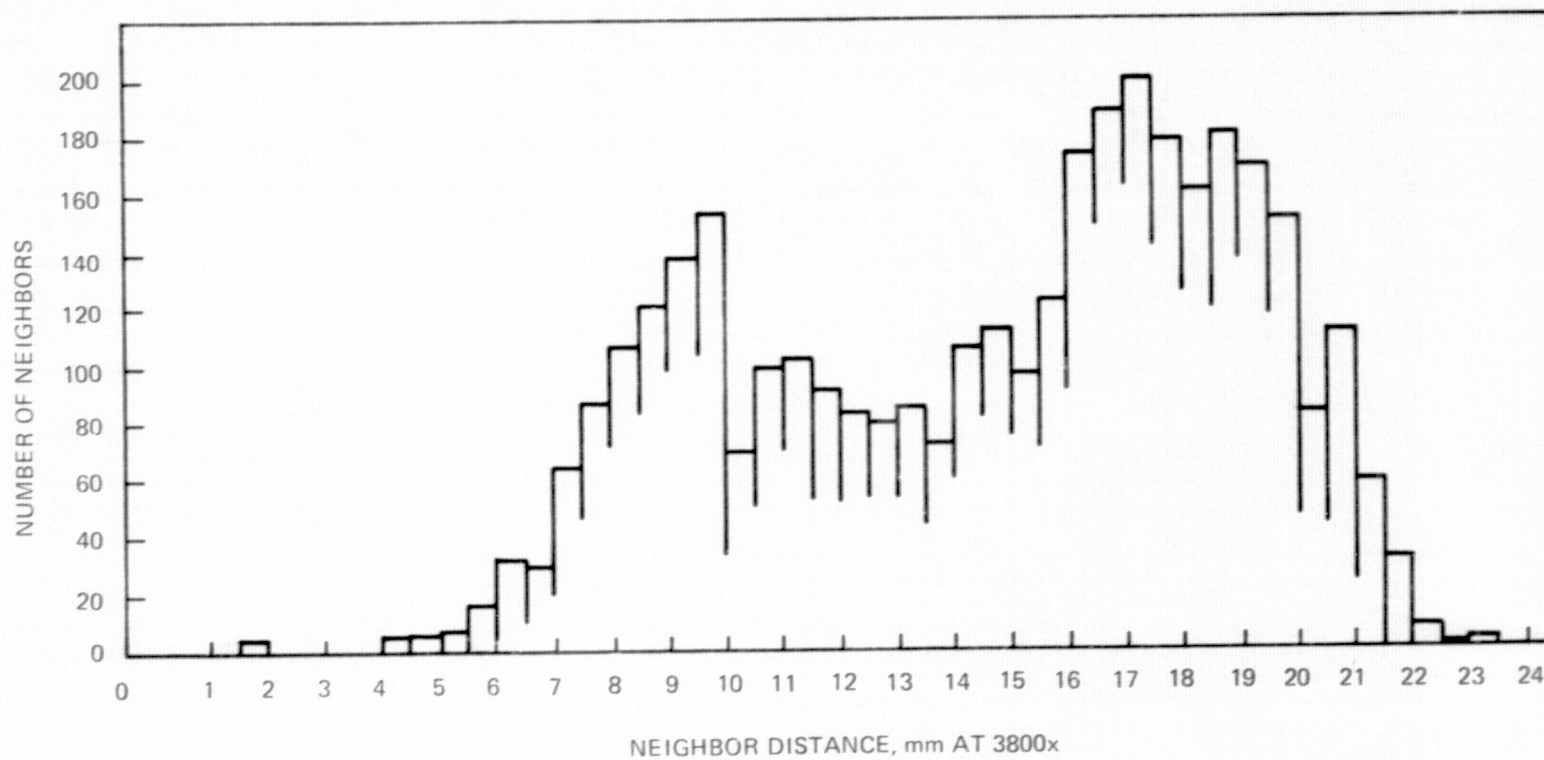


FIGURE 24. FIRST, SECOND, AND THIRD NEAREST NEIGHBOR DISTRIBUTION FOR Al-Al₃Ni EUTECTIC
SPECIMEN S75-26 #198; RATE 5 cm/hr; DATA BASE 198 POINTS

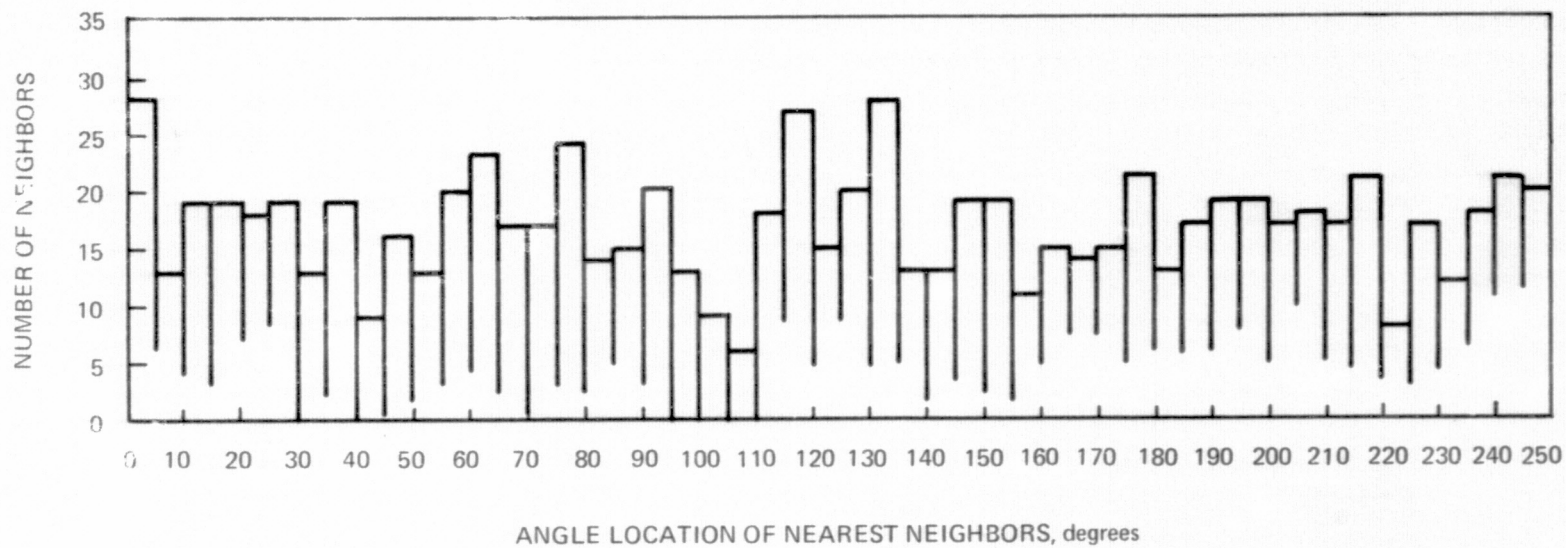
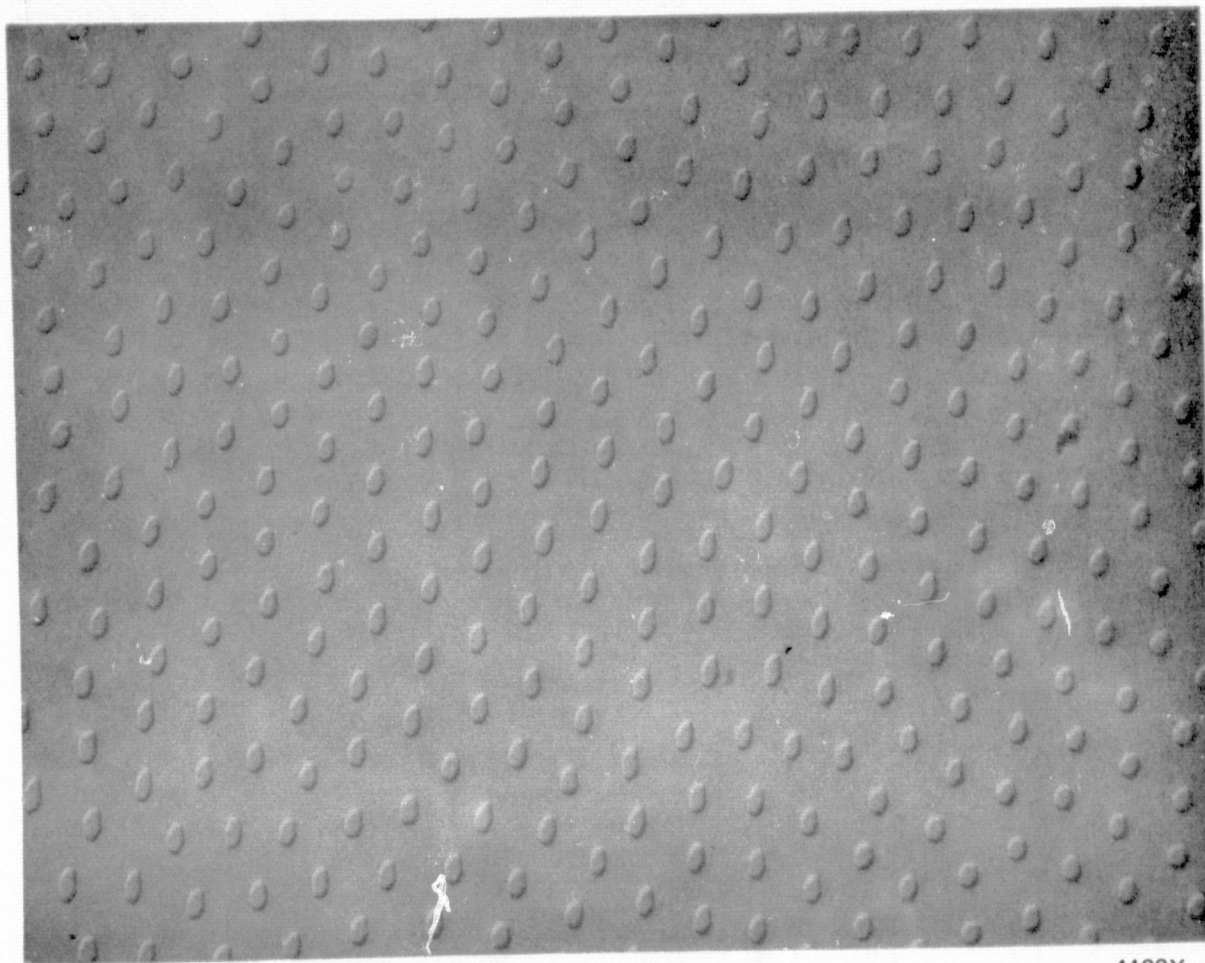


FIGURE 25. NEAREST NEIGHBOR ANGULAR DISTRIBUTION FOR Al-Al₃Ni

EUTECTIC SPECIMEN S75-26 #198; RATE 5 cm/hr; DATA BASE 198 POINTS



4400X

FIGURE 26. Al-Al₃Ni EUTECTIC SPECIMEN S75-28; REPLICA OF TRANSVERSE SECTION 4;
MAGNIFICATION 4400X; RATE 12 cm/hr

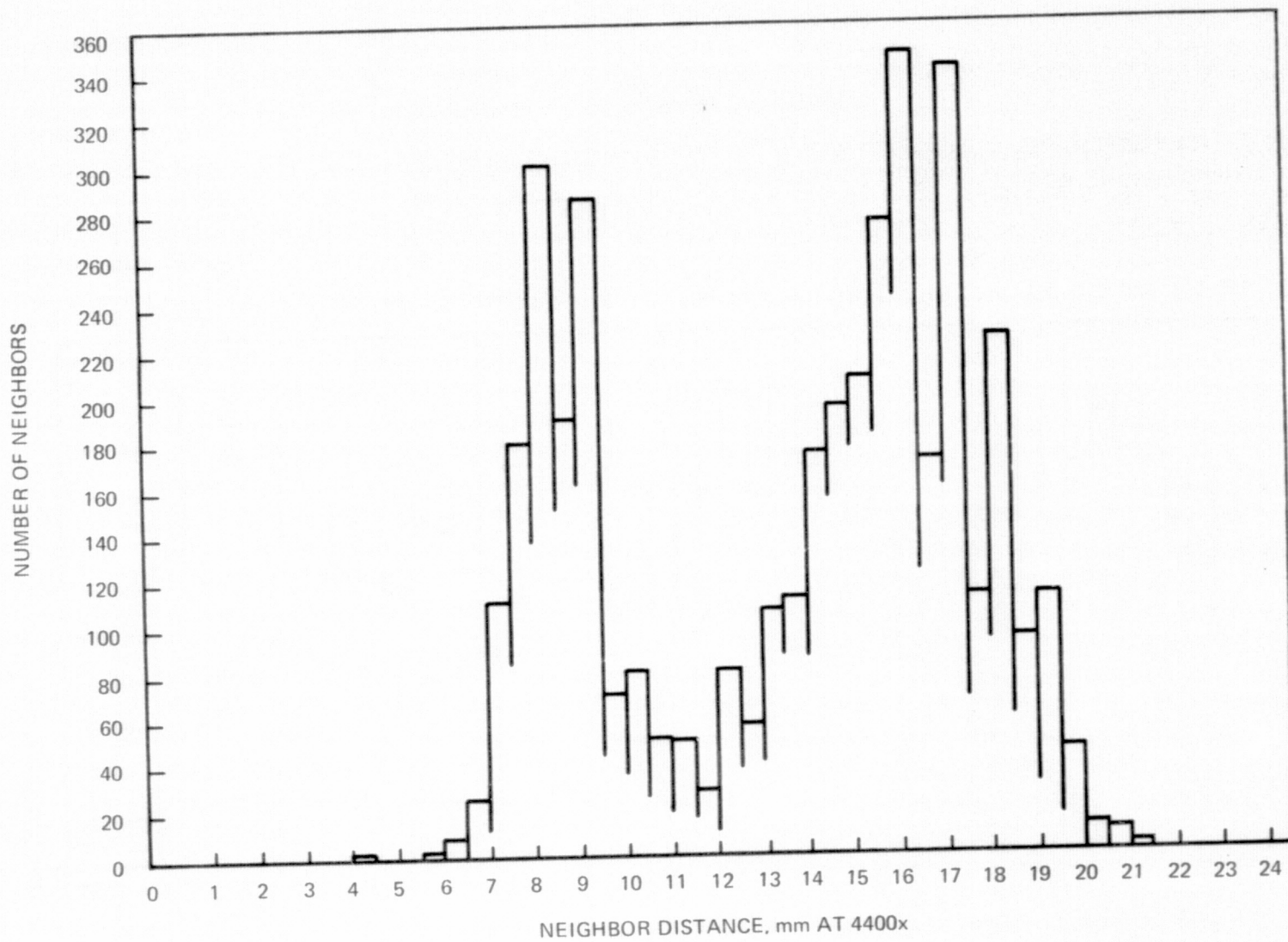


FIGURE 27. FIRST, SECOND, AND THIRD NEAREST NEIGHBOR DISTRIBUTION FOR Al-Al₃Ni EUTECTIC
SPECIMEN S75-28 #4; RATE 12 cm/hr; DATA BASE 226 POINTS

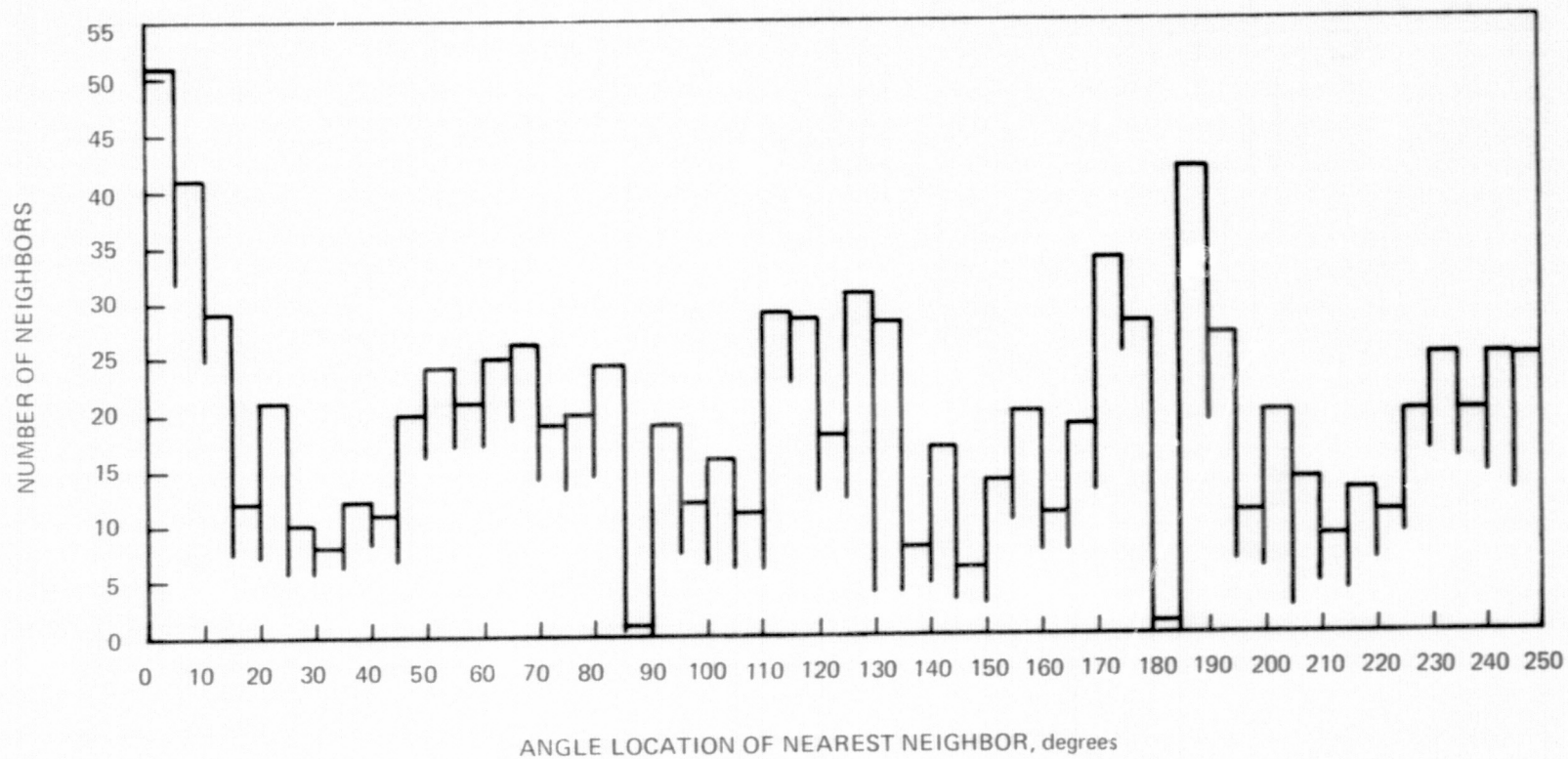


FIGURE. 28 NEAREST NEIGHBOR ANGULAR DISTRIBUTION FOR Al-Al₃ Ni EUTECTIC SPECIMEN S75-28 #4;
RATE 12 cm/hr; DATA BASE 226 POINTS

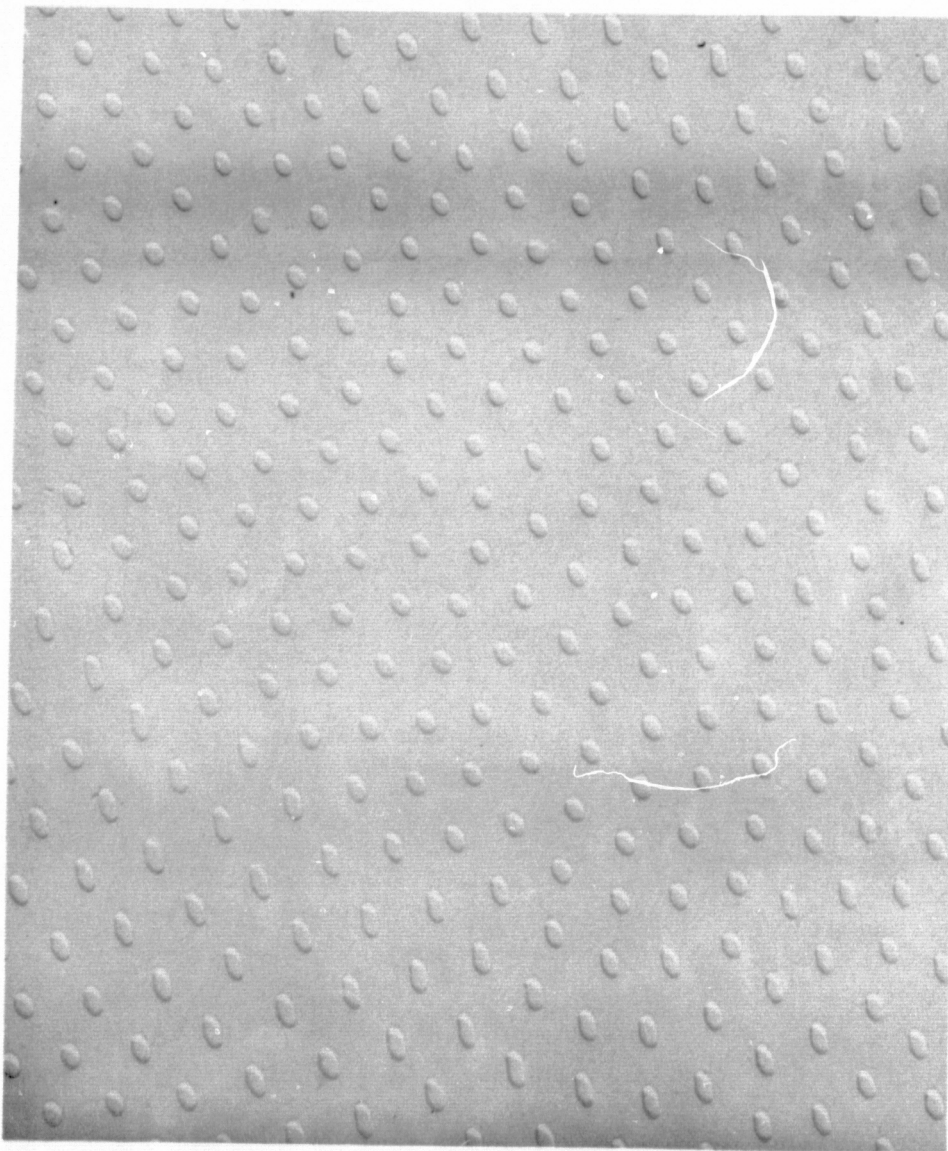


FIGURE 29. Al-Al₃Ni EUTECTIC SPECIMEN S75-28; REPLICA OF TRANSVERSE SECTION NO. 5; MAGNIFICATION 4400 X; RATE 12 cm/hr

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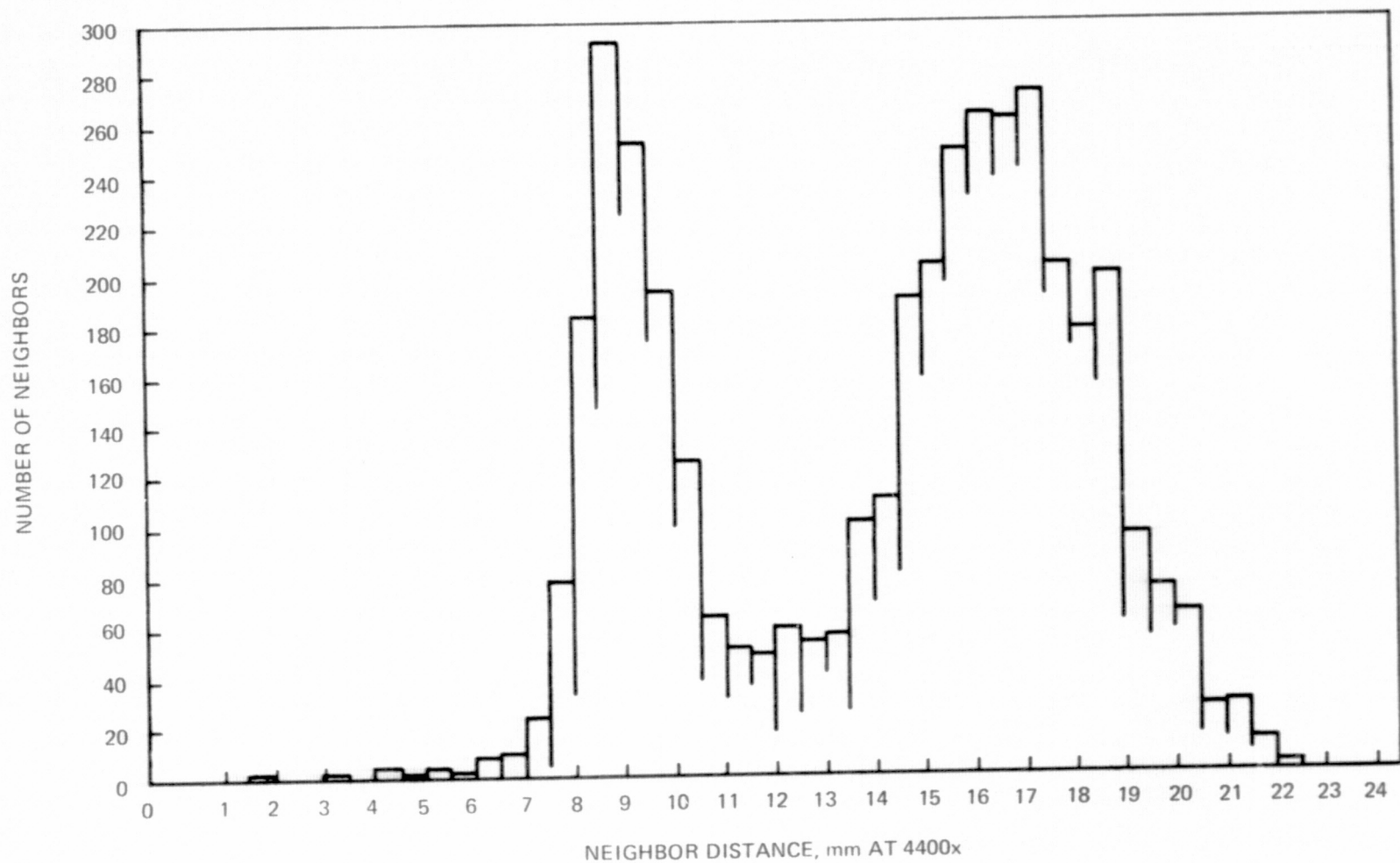


FIGURE 30. FIRST, SECOND, AND THIRD NEAREST NEIGHBOR DISTRIBUTION FOR Al-Al₃Ni
EUTECTIC SPECIMEN S75-28 #5; RATE 12 cw/hr; DATA BASE 227 POINTS

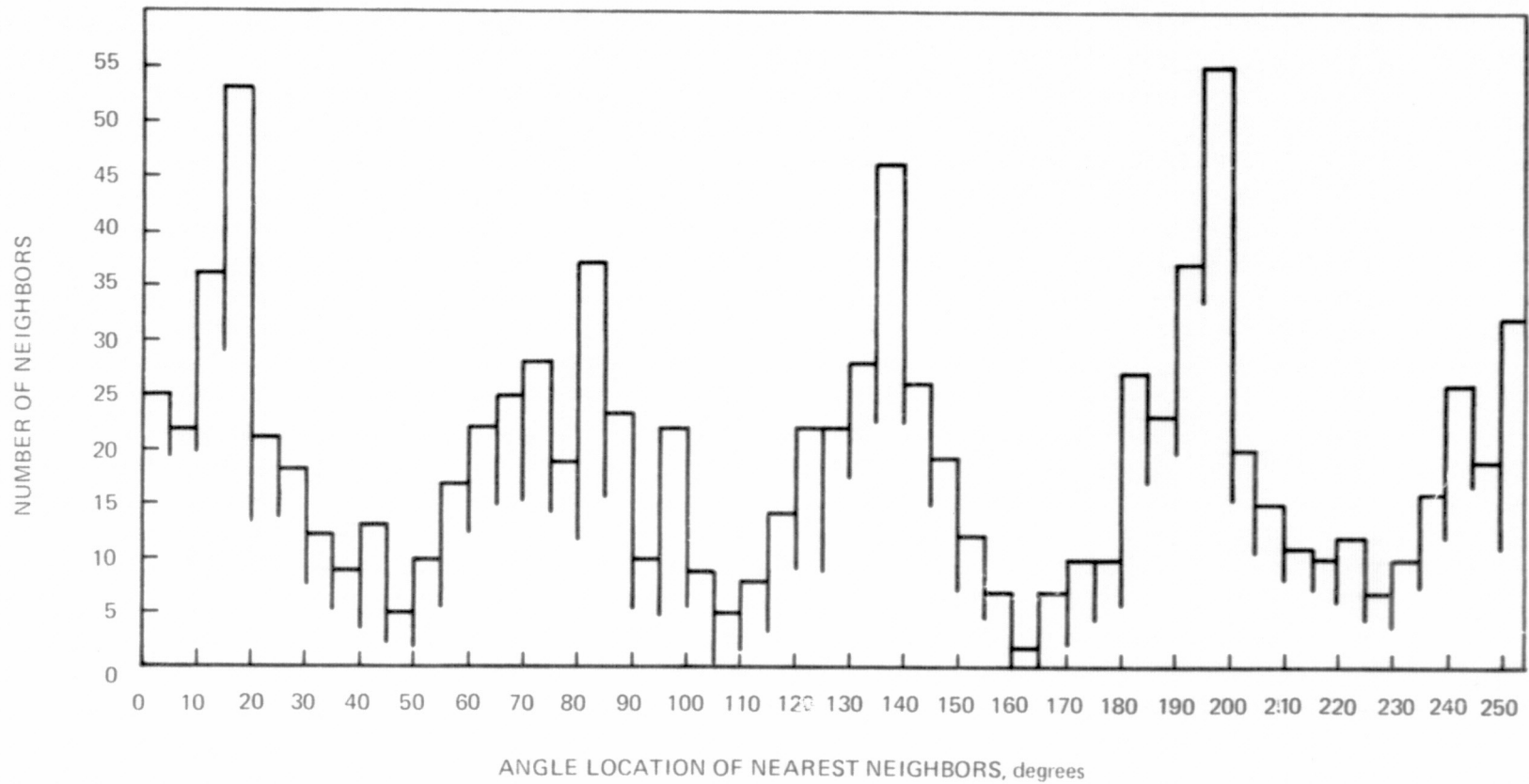
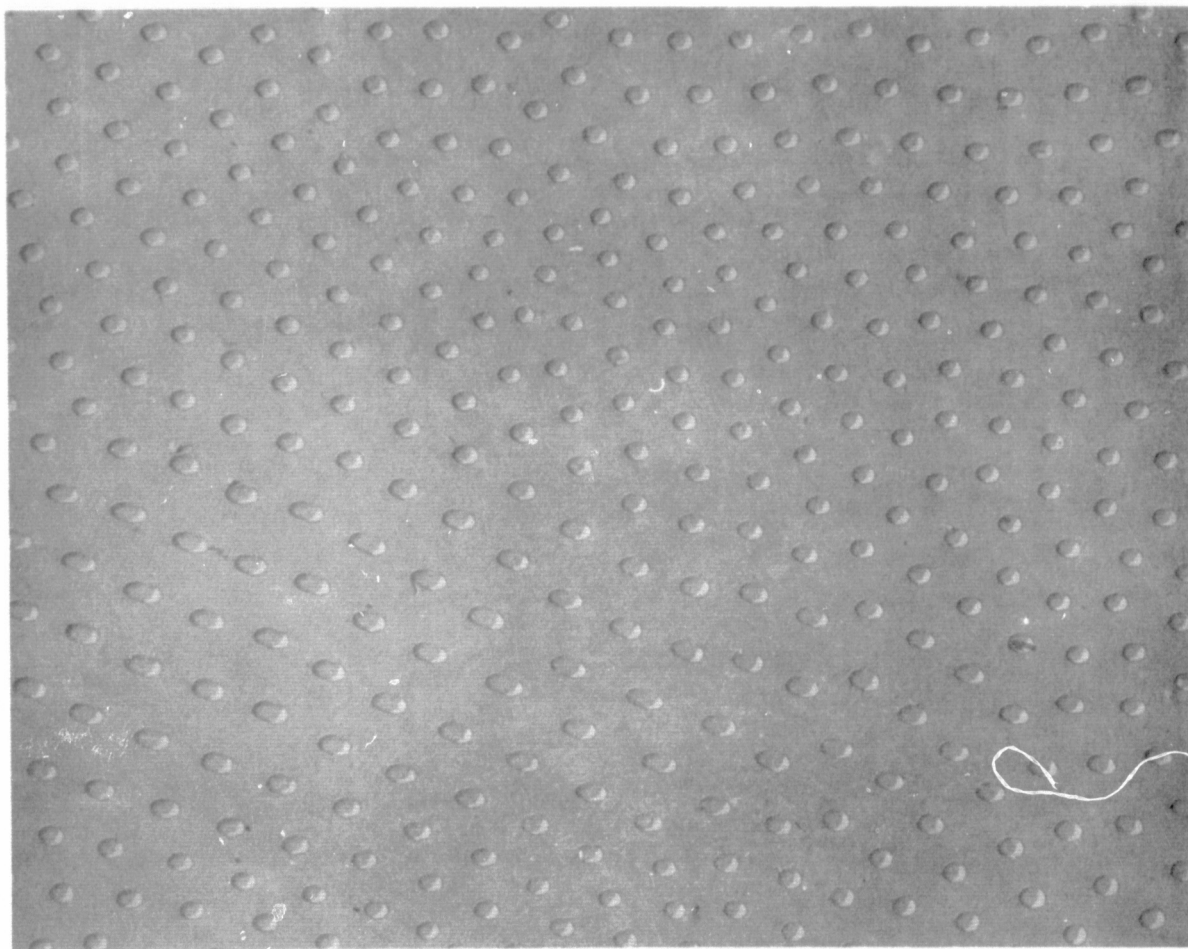


FIGURE 31. NEAREST NEIGHBOR ANGULAR DISTRIBUTION FOR Al-Al₃Ni EUTECTIC SPECIMEN

S75-28 #5; RATE 5 cm/hr; DATA BASE 227 POINTS



4400X

FIGURE 32. Al-Al₃Ni EUTECTIC SPECIMEN S75-29; REPLICA OF TRANSVERSE SECTION 2;
MAGNIFICATION 4400X; RATE 12 cm/hr

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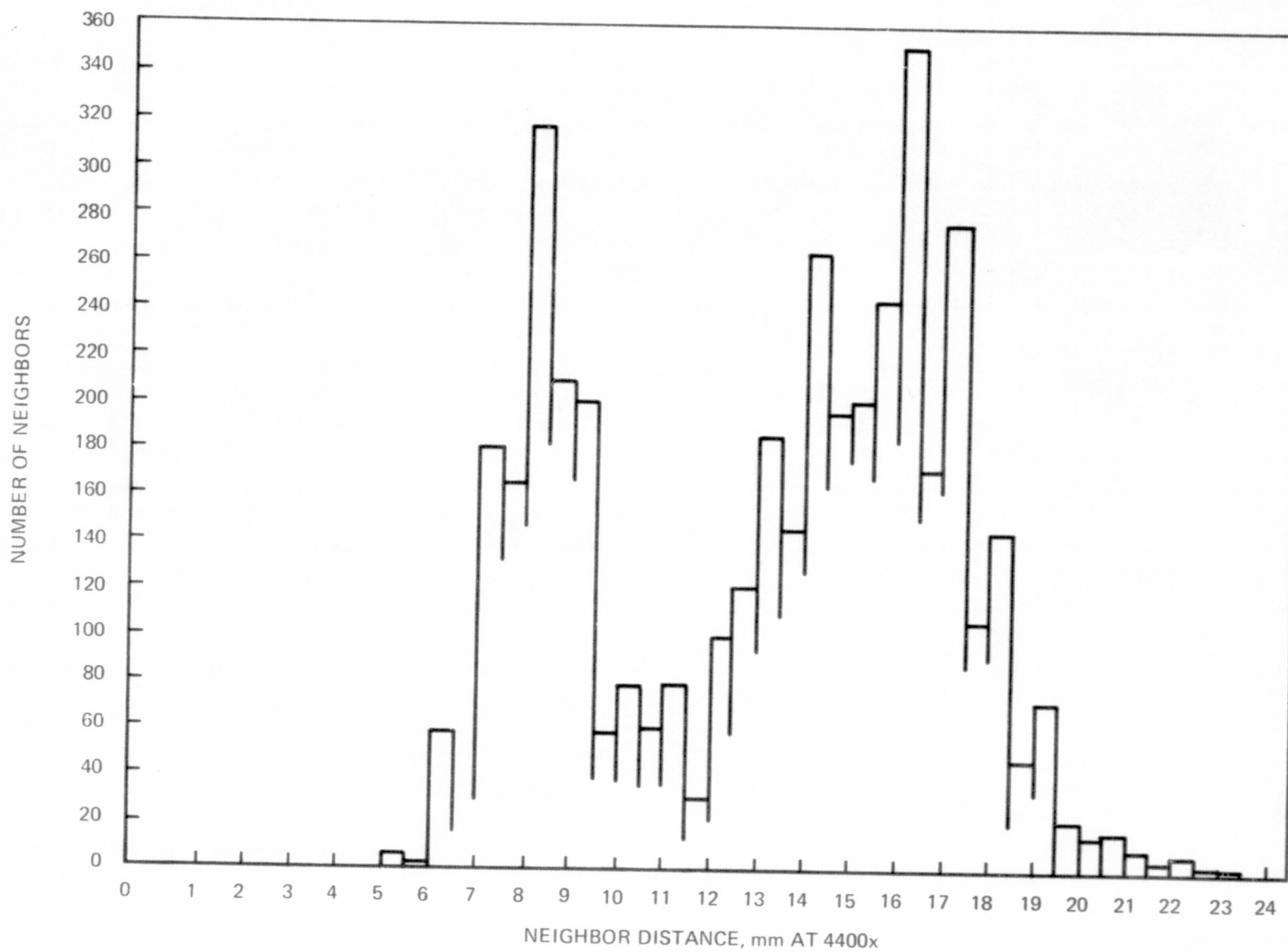


FIGURE 33. FIRST, SECOND, AND THIRD NEAREST NEIGHBOR DISTRIBUTION FOR Al-Al₃ Ni EUTECTIC
SPECIMEN S75-29 #2; RATE 12 cm/hr; DATA BASE 233 POINTS

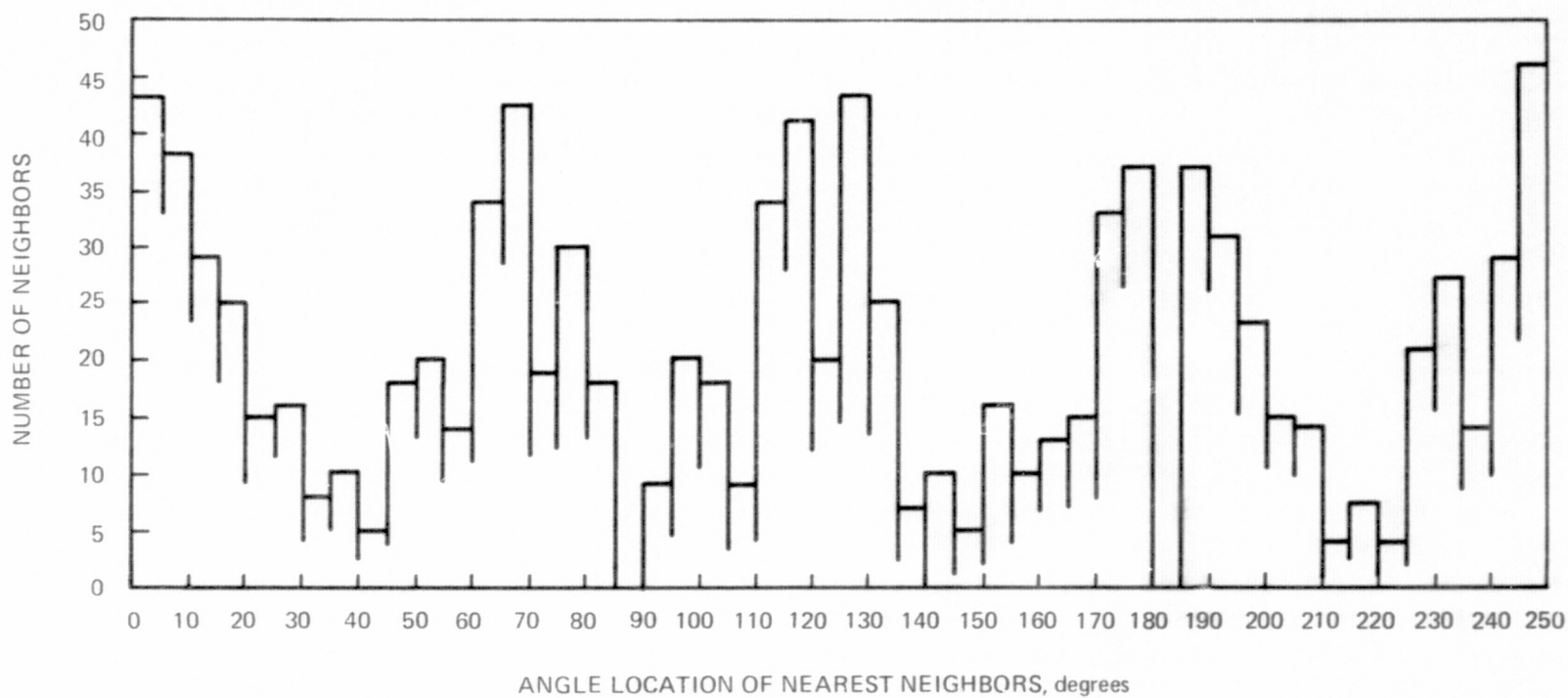


FIGURE 34. NEAREST NEIGHBOR ANGULAR DISTRIBUTION FOR Al-Al₃Ni EUTECTIC SPECIMEN

S75-29 #2 ; RATE 12 cm/hr; DATA BASE 233 POINTS

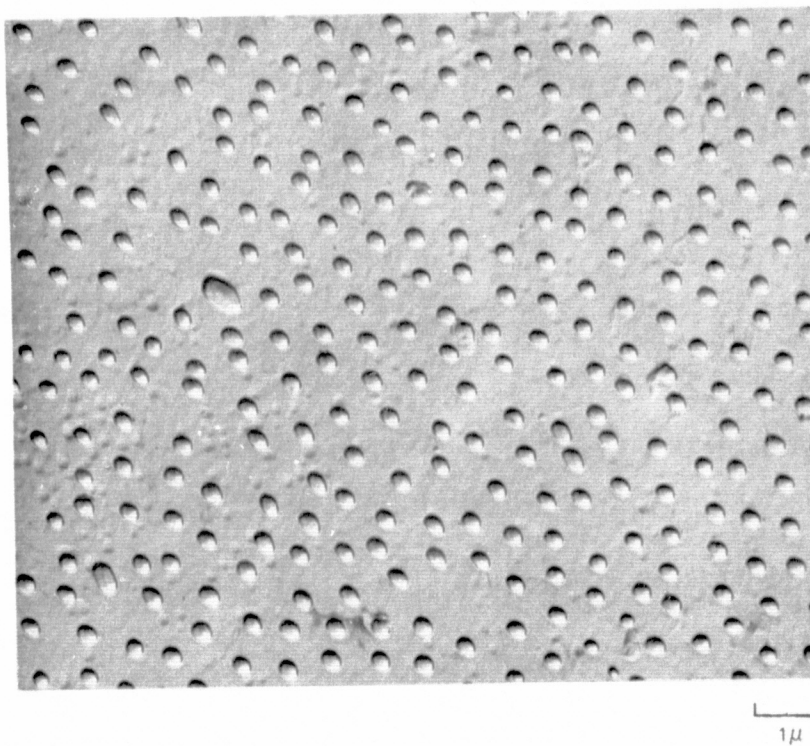
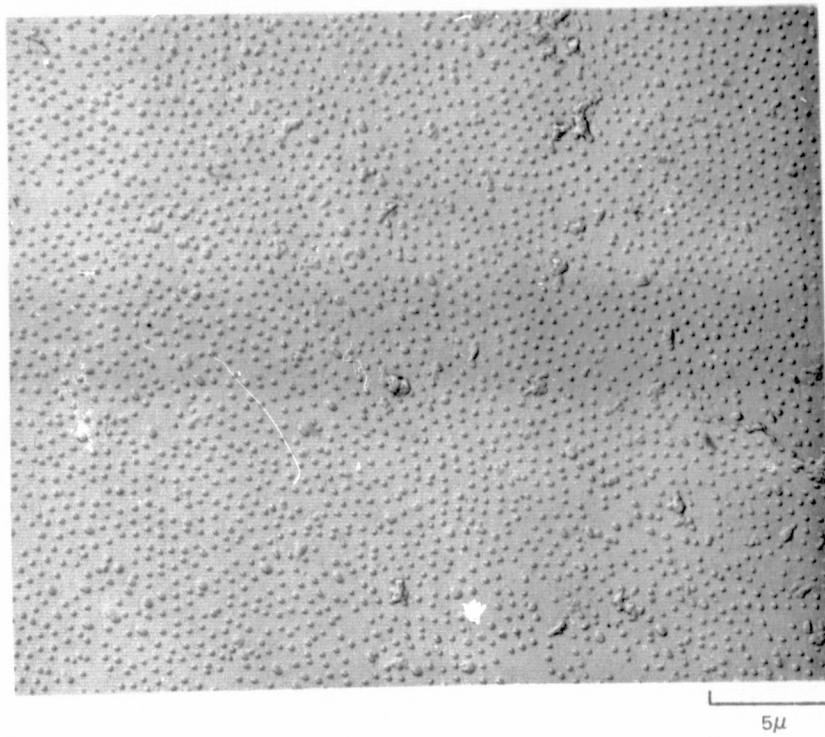


FIGURE 35. TRANSVERSE SECTION OF Al-Al₃Ni EUTECTIC SOLIDIFIED AT 100 CM/HR

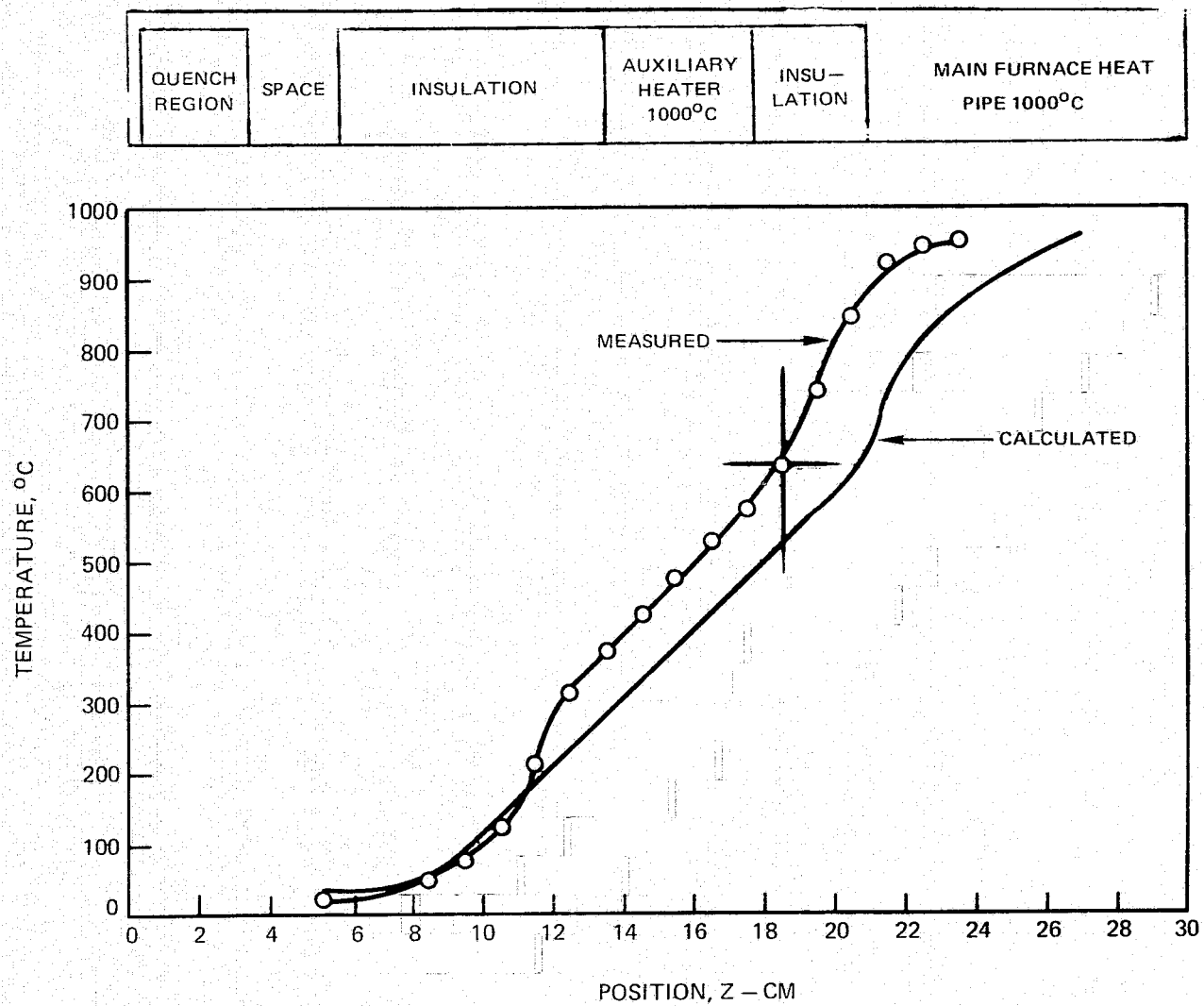


FIGURE 36. THERMAL PROFILES IN Al-Al₃Ni INGOT IN BRIDGEMAN FURNACE

APPENDIX I

```

CCC  PROGRAM TO COMPUTE GEOMETRY OF ROD-LIKE EUTECTICS
CCC
CCC
      DIMENSION X(800),Y(800),R(800),TE(800),INDEX(800)
      DIMENSION XX(800),YY(800),PR(800),XXX(800),YYY(800)
      DIMENSION JC(55), ME(75)
      ARAY = 4HARAY
      CALL IFILE(1,ARAY)
      NW = 0
      I=0
50    I=I+1
      READ(1,75), XXX(I),YYY(I),TM
75    FORMAT(2F,A3)
      NT=I
      IF(TM.EQ.'$') GO TO 135
      GO TO 50
135   TYPE 13,NT
13    FORMAT(1H,' THE NUMBER OF DATA POINTS IS ',2X,I4)
      DO 71 J=1,55
71    JC(J) = 0
      DO 72 J=1,75
72    ME(J) = 0
      CONTINUE
      TYPE 663
663   FORMAT(1H,' IS THE DATA IN MILLIMETERS ? ',)$)
      ACCEPT 667, MI
667   FORMAT(A1)
      IF( MI.EQ.'Y' ) GO TO 743
      DO 5 J=1,NT
5     XXX(J) = 10.*XXX(J)
      YYY(J) = 10.*YYY(J)
743   CONTINUE
      DO 85 J=1,NT
85    INDEX(J) = J
      X(J) = XXX(J)
      CONTINUE
      CALL SORT(NT,YYY,INDEX)
      DO 105 J=1,NT
105   L=INDEX(J)
      XXX(J) = X(L)
      CONTINUE
CCC
CCC  XXX,YYY ARE NOW SORTED ACCORDING TO INCREASING YYY VALUES
CCC
      TYPE 130
130   FORMAT(1H,' ENTER THE LOWER AND UPPER SCAN LIMITS ',)$)
      ACCEPT 145, SL,SU
145   FORMAT(2F)

```

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ORIGINAL DATA IS POOR

```

DO 695 K=1,NT
EX = XXX(K)
WYE = YYY(K)
IF (YYY(K) .LE. SL) GO TO 695
IF (XXX(K) .LE. SL) GO TO 695
IF (YYY(K) .GE. SU) GO TO 695
IF (XXX(K) .GE. SU) GO TO 695
DO 440 I=1,NT
X(I) = XXX(I) - EX
Y(I) = YYY(I) - WYE
440 CONTINUE
DO 160 J=1,NT
P(J) = SQRT( X(J)**2 + Y(J)**2 )
INDEX(J) = J
160 CONTINUE
CALL SORT(NT,R,INDEX)
DO 190 I=1,NT
L = INDEX(I)
Q = Y(L)/X(L)
TE(I) = ATAN(Q)*180./3.14159
IF( X(L) ) 10,190,15
10 TE(I) = TE(I) + 180.
GO TO 190
15 IF( Y(L) ) 20,190,190
20 TE(I) = TE(I) + 360.
190 CONTINUE
CALL WINDOW(P,TE,JC,ME)
NW = NW+1
TYPE 66,NW
66 FORMAT(1H,2X,13)
695 CONTINUE
TYPE 675
675 FORMAT(1H,' POSITION POINT COUNT')
TYPE 200,(I,JC(I),I=1,51)
200 FORMAT(1H,3X,13,5X,16)
TYPE 680
680 FORMAT(1H,/, ' POSITION ANGLE COUNT')
TYPE 220,(I,ME(I),I=1,73)
220 FORMAT(1H,3X,13,5X,16)
380 STOP
END
CCC
SUBROUTINE SORT(NT,A,INDEX)
DIMENSION A(200),INDEX(200)
M = NT
1 M = M/2
IF( M .EQ. 1 ) RETURN
K=NT-M
J=1
2 I=J
3 IM=I+1
IF( A(I) - A(IM) ) 5,5,4
4 SAV = A(I)

```

```

A(I) = A(IM)
NSAV = INDEX(I)
INDEX(I) = INDEX(IM)
A(IM) = SAV
INDEX(IM) = NSAV
I = I - M
IF ( I .GE. 1 ) GO TO 3
5 J = J + 1
IF (J-K) 2,2,1
END

CCC
SUBROUTINE WINDOW(R,TE,JC,ME)
DIMENSION R(800),TE(800),JC(55),ME(75)
OP = 0.5
DO 4 L=1,19
DO 3 K=1,50
WIN = OP*FLOAT(K)
IF( R(L) .LE. WIN ) GO TO 1
3 CONTINUE
1 JC(K) = JC(K) + 1
4 CONTINUE
NP = 5
DO 16 L = 2,7
DO 15 K = 1,73
MM = NP*K
DOW = FLOAT(MM)
IF( TE(L) .LE. DOW ) GO TO 18
CCC - IF TE - DOW IS +, TE HAS NOT BEEN FOUND; MOVE POINTER
CCC IF TE - DOW IS 0 OR -, TE HAS BEEN FOUND; COUNT AND
CCC ADVANCE L TO THE NEXT NEAREST NEIGHBOR
15 CONTINUE
18 ME(K) = ME(K) + 1
16 CONTINUE
RETURN
END

```

APPENDIX II

Prior to the program for Processing Eutectics in Space, the SINDA program had not been used extensively in the Research Center. Those features of SINDA, in the form of subroutines, used to account for nonlinear thermophysical properties and the phase change energy had never been used. Therefore, it was necessary to spend some time verifying the program and especially these features. Ice water systems were used in this phase of the program.

Several runs were made involving one, two and three dimensional geometries and all forms of boundary conditions. These data were reported in Ref. AII-1. All values of thermophysical properties for the verification calculations were taken from Ref. AII-2.

The thermal conductivity of the water in the liquid state was taken from Ref. AII-3.

A point to be noted is no allowance was made for volume changes as a function of temperature change. The effect caused by this should be of secondary importance and could be reflected in a pseudo-thermal conductivity and specific heat if one wanted greater accuracy. The SINDA program was then used to examine the experimental furnace shown in Fig. AII-1.

The thermal model made with SINDA divided the 40 cm long experimental system ingot (Fig. AII-1) into 100 vertical nodes for each of five radial positions for a total of 500 internal nodes (Fig. AII-2). The physical breakup is shown for this system on Fig. AII-3 for a typical section. Parameters for the material being processed in the first model were those of the Al-Al₃Ni eutectic contained in a thin-walled aluminum oxide tube. The material thermophysical properties are listed on Figs. AII-4 through AII-8. Transient analyses were performed on this system with the boundary conditions given on Fig. AII-1. Running times were long, approximately 30 min machine time, because the system was constructed to show the shape of the interface and not just its axial location. The breakup used for the radial direction was done to produce even radial increments. This led to small interior volumes and small conduction surfaces on the inner elements.

At this point in the program, it was decided to use other options in SINDA so that steady state solution could be obtained for the various systems under consideration. The phase change energy does not enter into these computations and, of course, no times are considered. The final location of the interface is uniquely determined, however, and the answers are important as a comparative tool in the studies of furnace parameters.

Studies on the SINDA thermal analysis program indicate that the approach to steady-state heat flow in the model was asymptotic, thus requiring a large number of computing cycles, and relatively long run times for them. It was estimated that adequate convergence would be reached after $2(10)^4$ iterations in the program. It should also be noted that the use of the SINDA program as a tool for evaluating changed configurations of a given model is hindered by the present input form requiring complete reinsertion of node specification for even minor changes in the model.

The leaky insulation in the model is 5 cm of fiberfax ($R = 0.86 \text{ BTU/m ft}^2 \text{ } ^\circ\text{F/in.}$). Heat leakage is allowed only in the radial direction. The emissivity (ϵ) of the Al_2O_3 was taken to be 0.6 and the Inconel thermal liner on the heater was taken as 1.0, i.e., black body, for computing the radiation coefficients used in SINDA. The heat transfer coefficient in the quench region was taken as 2000.

References

- AII-1. Douglas, F. C. and F. S. Galasso: Processing Eutectics in Space. Interim Report, Contract NAS8-29669-S/A2, Dec. 1974.
- AII-2. Sears, F. W. and M. W. Zemansky: University Physics. Addison Wesley, 1964.
- AII-3. Rohsenow, W. and H. Choi: Heat Mass and Momentum Transfer. Prentice Hall, 1961.

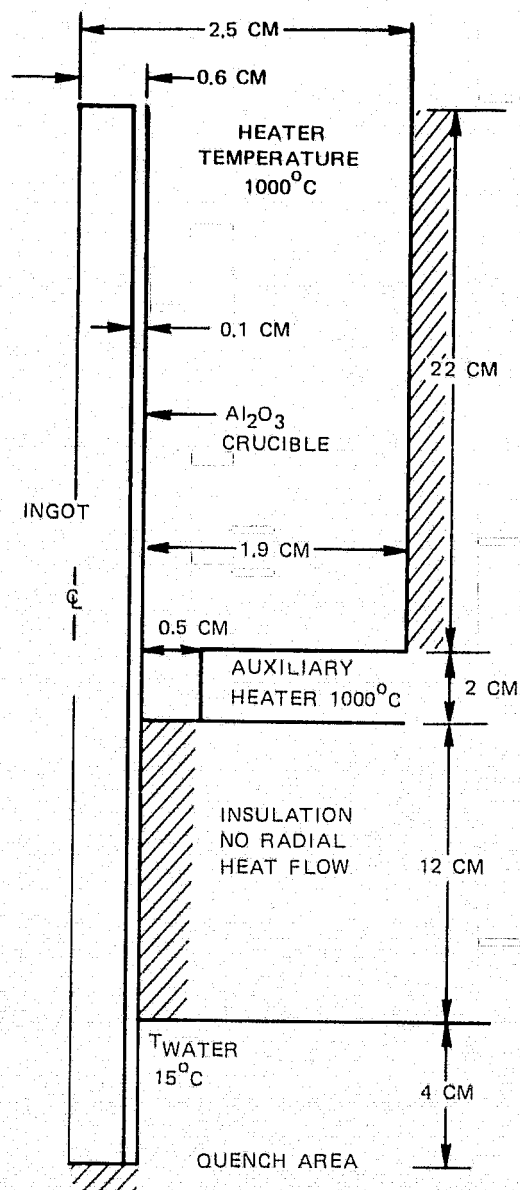


FIGURE AII-1. SYSTEM MODELS USED FOR SINDA THERMAL ANALYSIS



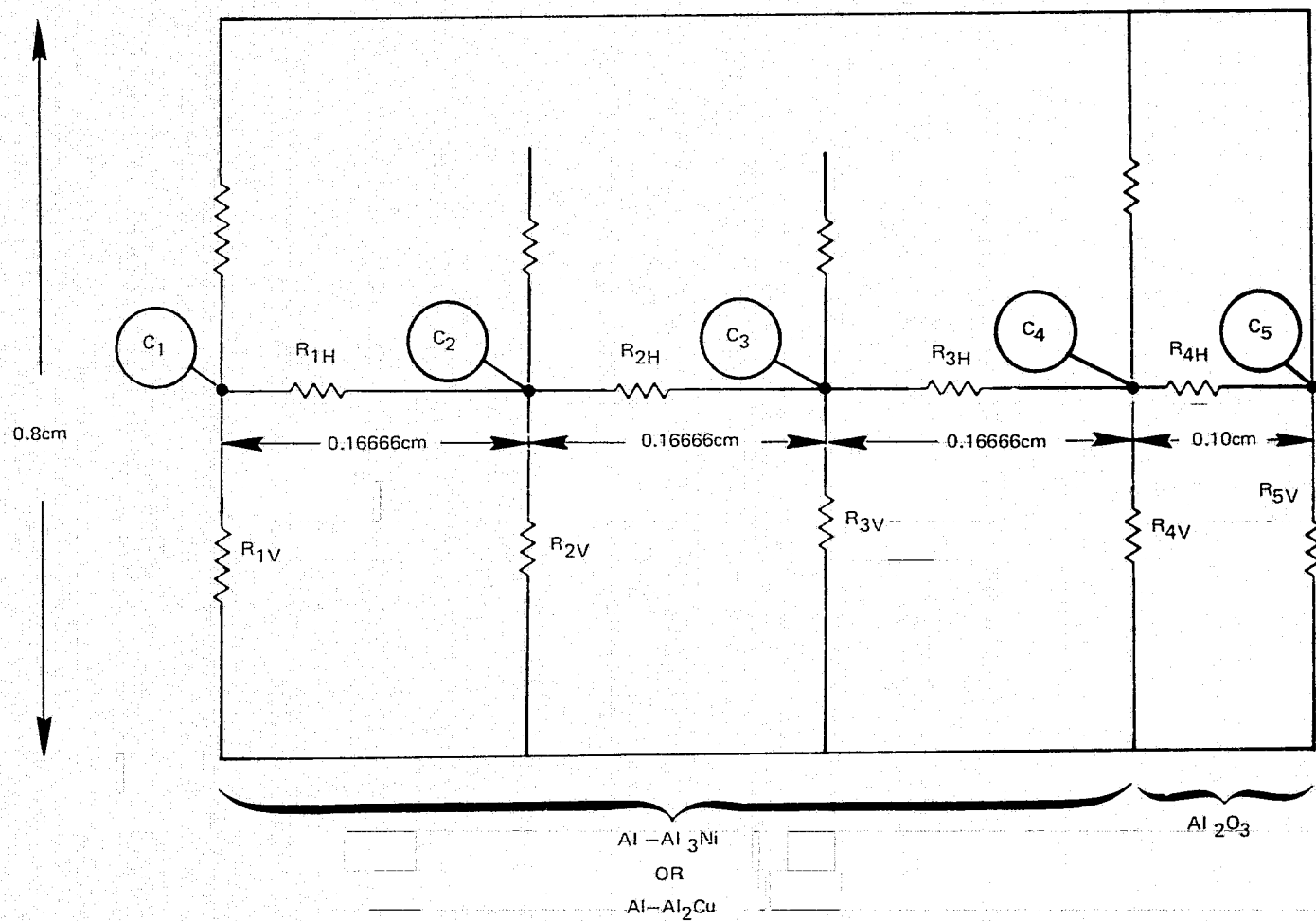


FIGURE AII-3 PHYSICAL GRID POINT POSITION

SINGLE OXIDE/ALUMINUM

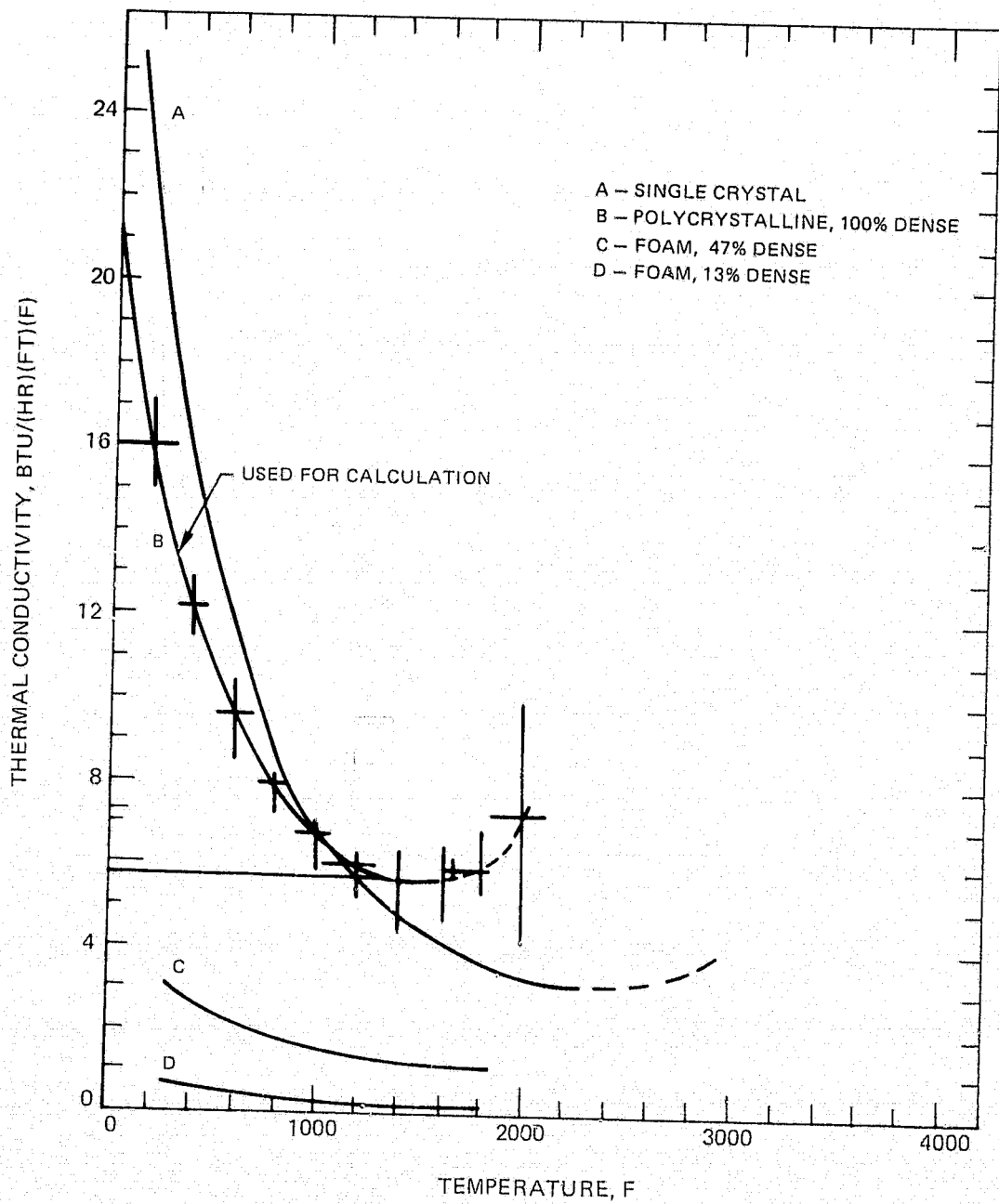
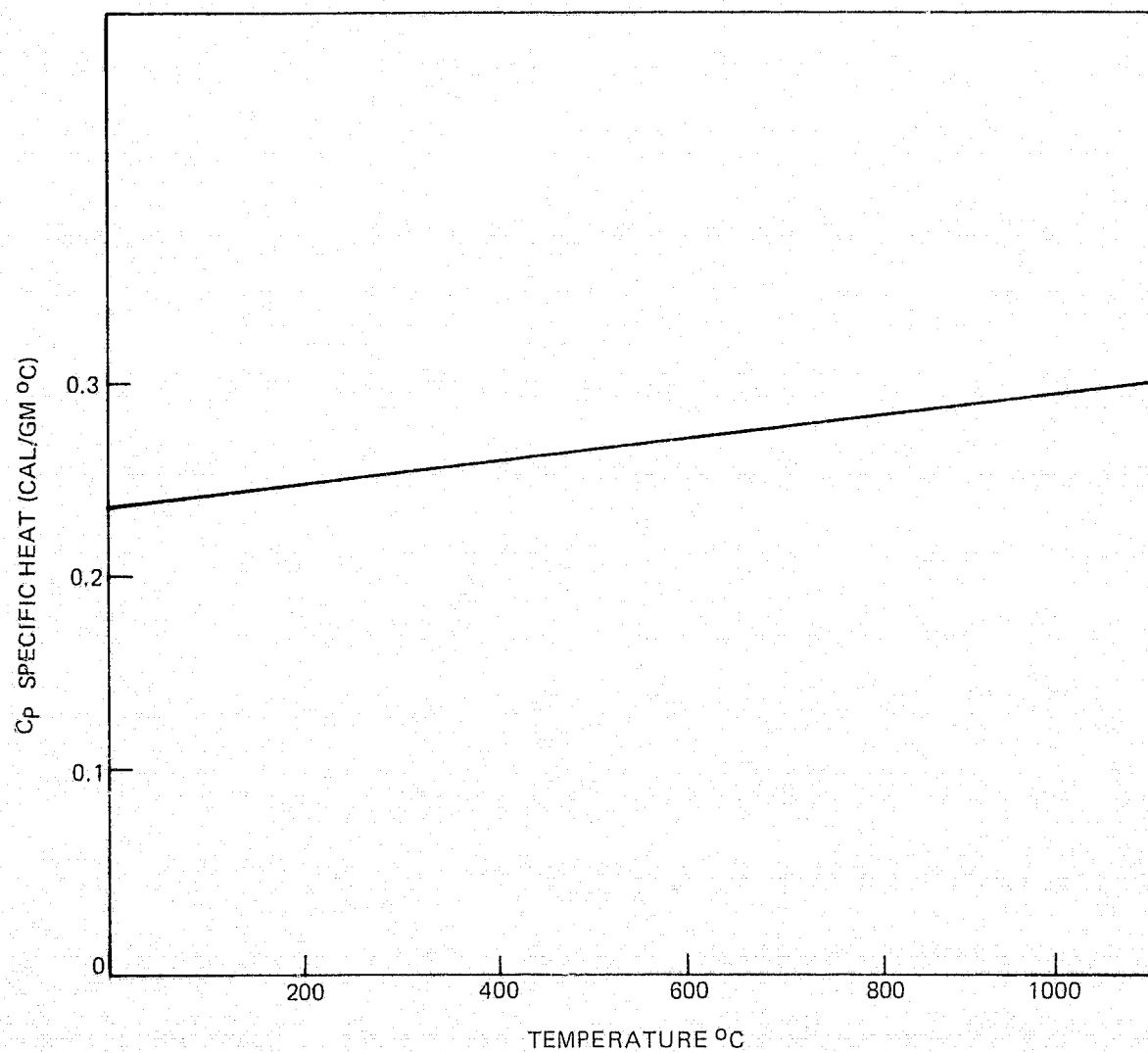
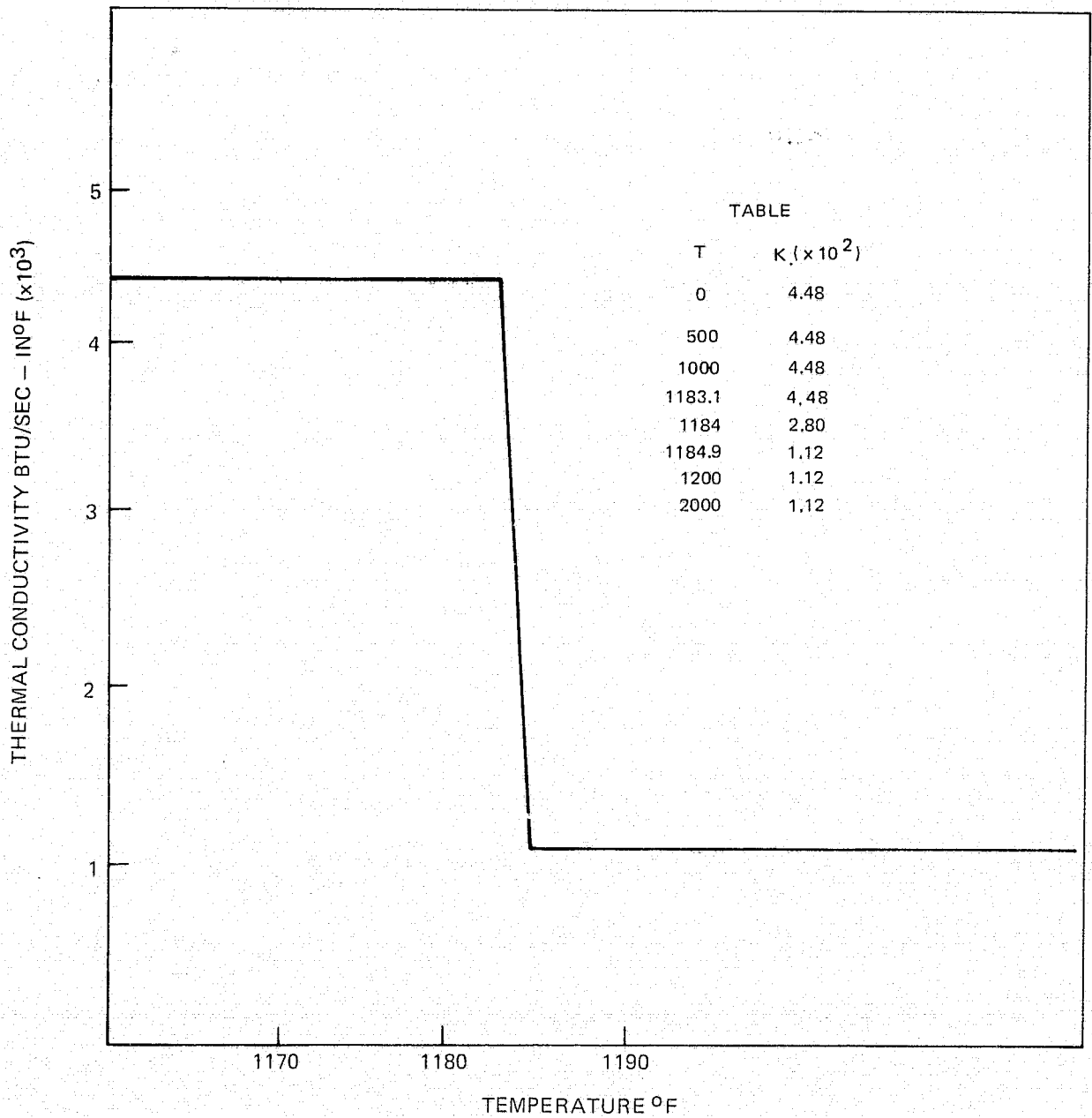


FIGURE AII-4. THERMAL CONDUCTIVITY OF SINGLE-CRYSTAL AND POLYCRYSTALLINE ALUMINA

REF. ENGINEERING PROPERTIES OF SELECTED CERAMIC MATERIALS PUBL. AM. CERAM. SOC. 1966

FIGURE AII-5. Al_2O_3 SPECIFIC HEAT (CAL/GM °C)

FIGURE AII-6. THERMAL CONDUCTIVITY OF EUTECTIC Al-Al₃Ni

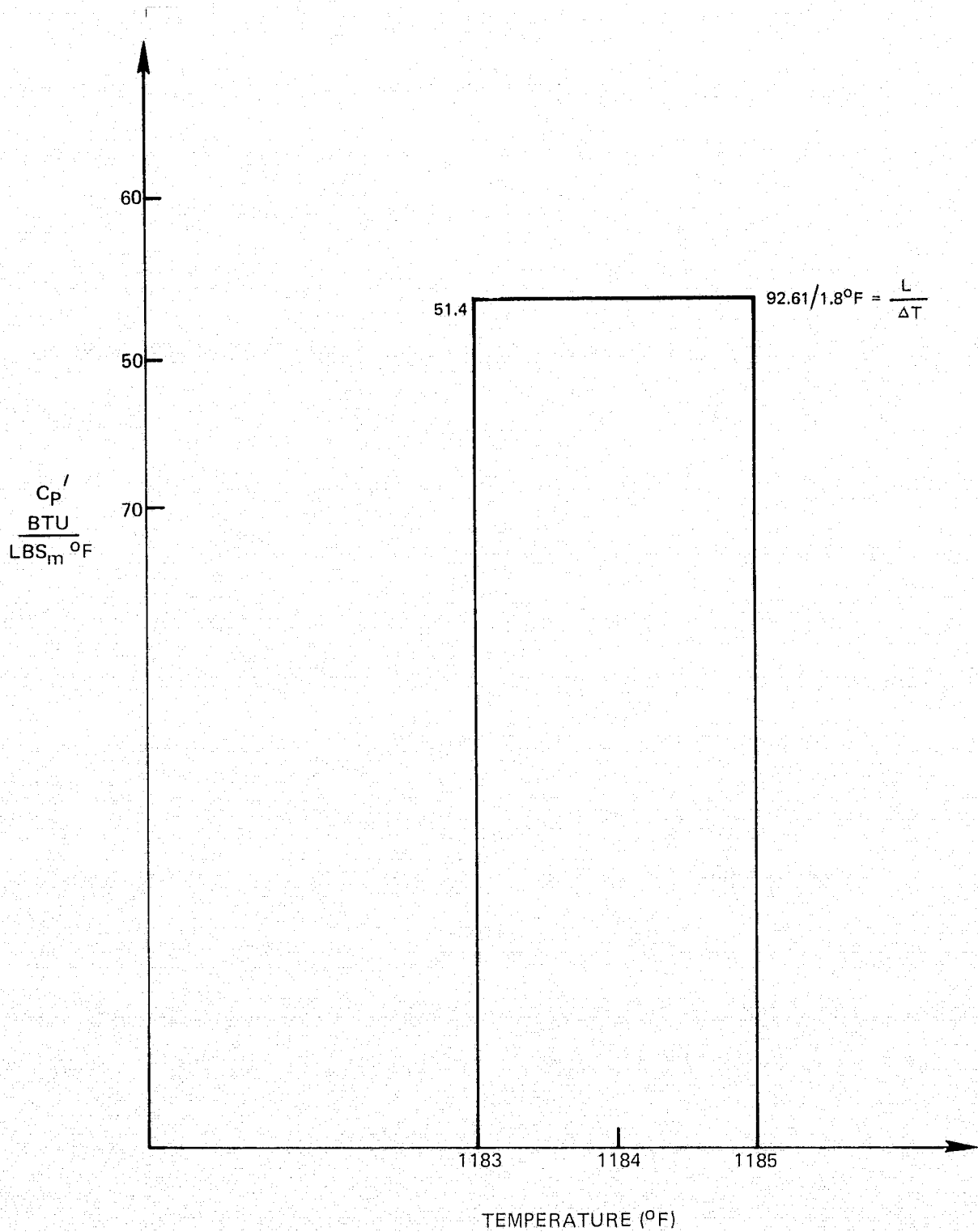
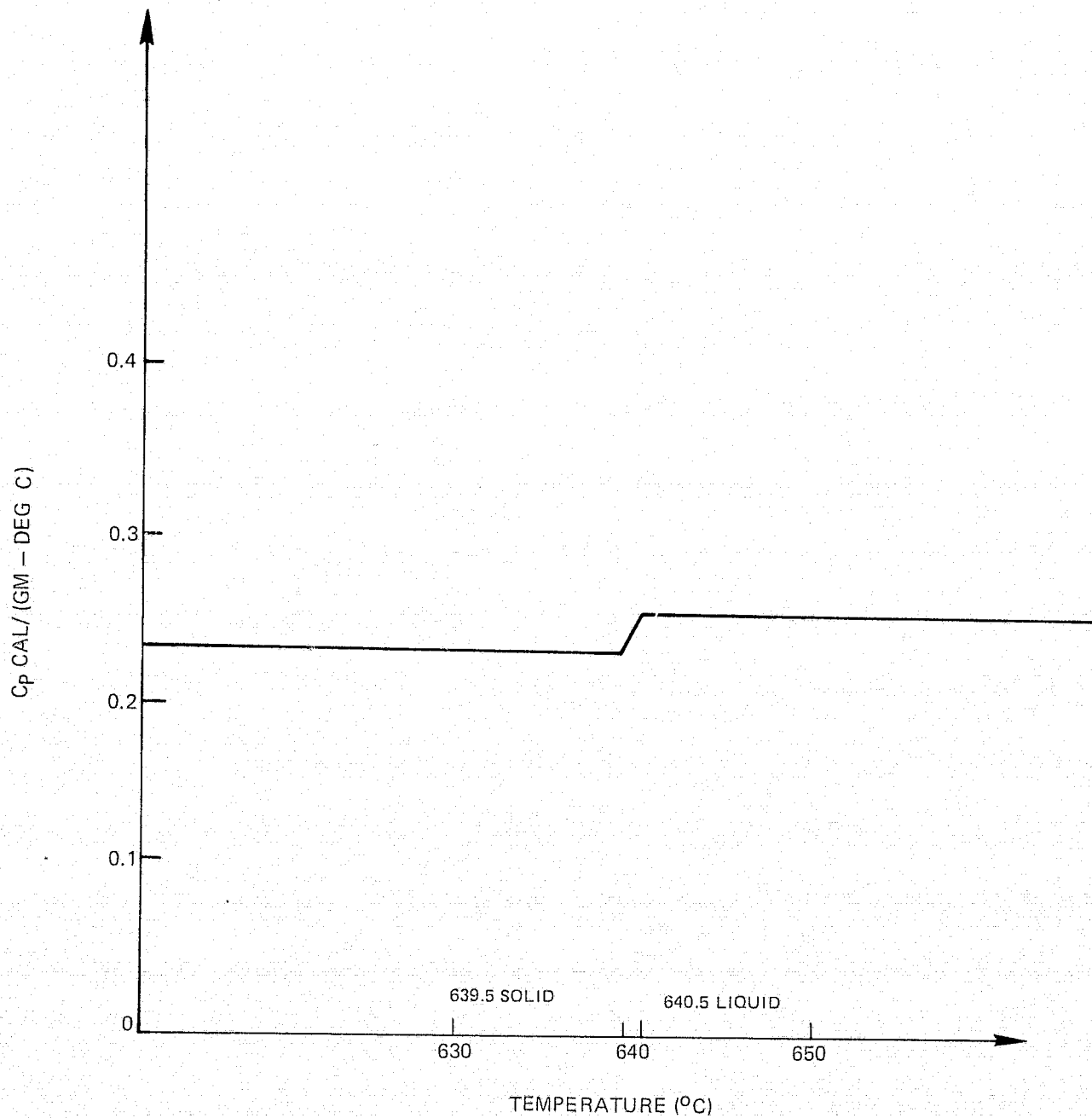


FIGURE AII-7. LATENT HEAT VS TEMPERATURE Al-Al₃Ni

FIGURE AII-8. Al-Al₃Ni SPECIFIC HEAT (CAL/GM °C)